

# **NUCLEAR STANDARDS** for Chemistry and Technology





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## NUCLEAR STANDARDS for CHEMISTRY and TECHNOLOGY

## NUCLEAR STANDARDS FOR INDUSTRY, SCIENCE, GOVERNMENT and CONSUMER

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Proceedings of a Symposium on Standards in Nuclear Chemistry and Technology held at the 156th National Meeting of the American Chemical Society, Atlantic City, New Jersey September 8 - 9, 1968



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## ABSTRACT

Many diverse organizations are engaged in developing the nuclear standards needed by science, industry, and government for the rapidly growing field of nuclear technology. Often there has been inadequate communication between these groups and insufficient opportunity to get an overall picture of the needs of the country for the many different kinds of standards necessary for this new industry. The Symposium sponsored by the American Chemical Society Division of Nuclear Chemistry and Technology in cooperation with the Committee on Standardization Relations brought together most of the groups with interests in standards for nuclear chemistry and technology to identify the problems, to discuss present programs, and to outline future needs for standards in this broad field. Standards for purposes of the symposium were defined very broadly to include standard materials, standard procedures, standard specifications, standard data, and engineering standards. The mechanisms available for standardization, the "state of the art" in standardization in many areas of the nuclear field, some indications of unmet needs, and estimates of future needs were discussed in a series of papers and panel discussions which are presented here in either complete or summary form.

KEY WORDS: activation analysis standards, isotope reference standards, nuclear fuel burn-up standards, radiation effect standards, radioactivity standards, standards for nuclear chemistry, standards for nuclear technology.

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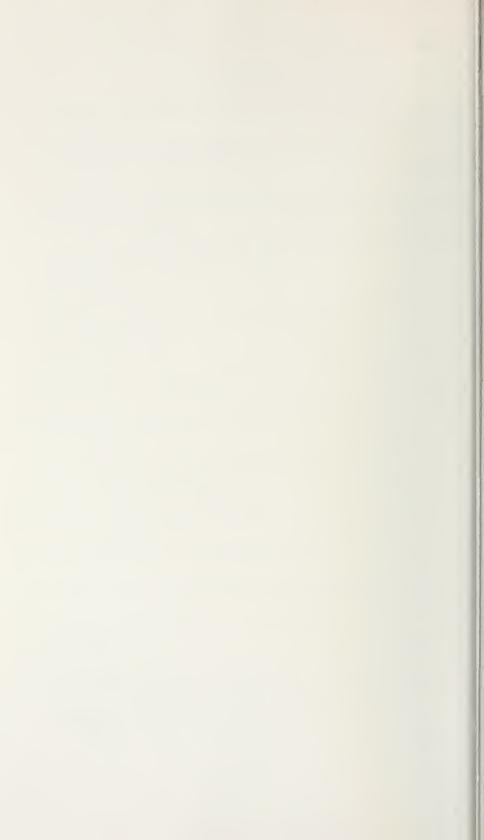
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## Introduction

W. Wayne Meinke and Hugh F. Beeghly The continuing rapid development of uses for nuclear energy in research, in medicine, in science and for generating power is making the development of standards imperative in the diverse areas of safety, health, quality assurance, and specific engineering standards. This is a significant sign of the growing maturity of the applications of nuclear energy in chemistry and industrial technology.

There are many diverse organizations and groups involved in developing nuclear standards in response to the recognized needs of science, industry and government. Often, however, there has been little communication between these groups and very little opportunity to get an overall picture of the needs of the nation for standards, or to obtain authoritative knowledge of the mechanisms available for standardization.

The very rapid development and expansion of the nuclear power industry has highlighted especially the need for specific engineering and materials standards, and for reliable methods for making meaningful measurements of burn-up, fall out, atmospheric contamination, and radiation effects on materials, in order to maintain the outstanding safety record which has been established. Equally important are standards for monitoring and for accurately measuring radiation in chemical processing, medicine, and research.

The Symposium on Standards for Nuclear Chemistry and Technology, sponsored by the American Chemical Society Division of Nuclear Chemistry and Technology in cooperation with the ACS Committee on Standardization Relations was planned to bring together the many groups having interests in standards for nuclear chemistry and technology, and to discuss present programs and future needs for such standards. Standards, for purposes of the Symposium, were defined very broadly to include standard procedures, standard specifications, standard materials, standard data, and engineering standards.

### Introduction

The ACS, unlike a number of societies, does not have a formal program for considering and approving standards. Recognizing this, the Council Policy Committee voted in 1955 to establish a Committee on Standardization Relations, reporting to the Council, "which will consider and make recommendations on all proposals that the Society engage in standardization activities or cooperate in such projects and which also will attempt to obtain comments on chemical standards submitted to the ACS for review." Furthermore, this Committee was to be, and is, separate from ACS activities on atomic weights, nomenclature, analytical reagents and air pollution.

The Committee has served the two-fold purpose of keeping the ACS, as an organization, out of direct involvement in standardization, and of assuring that needs of ACS members for standards are adequately cared for through appropriatelyestablished standardization bodies. It serves as a clearinghouse and information center for standards problems of the Society's members and has referred to it standards matters that are submitted to the Society.

The ACS has official representation on many standardizing organizations. For example, the ACS is represented on USASI Standards Boards and also has official representatives on a number of the individual committees within these Boards. These representatives, who report to the Council, function to keep the Society informed on standards efforts in these areas and to alert the ACS membership to potential standards problems and needs as they arise. They report, at least annually, to the ACS Council and their reports are published periodically in Chemical and Engineering News.

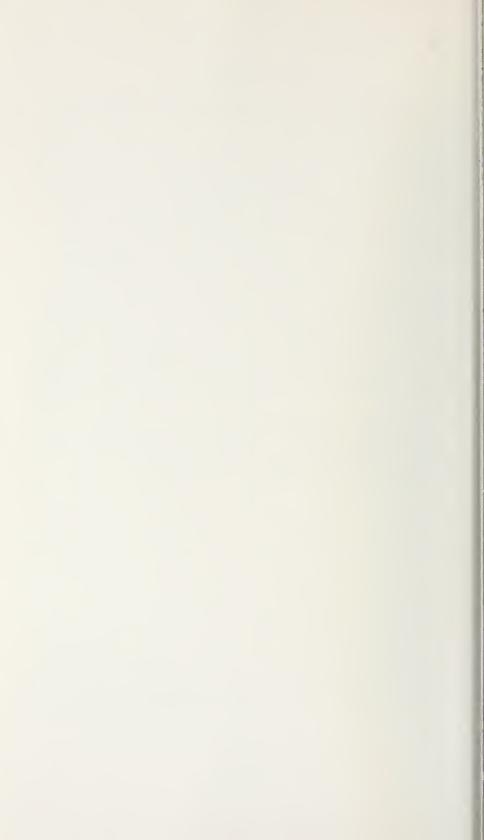
The ACS representation, as a Society, on ASTM Committees, is less formal, but the Society is well represented through individual membership of ACS members on Committees, Task Forces, District Councils and Advisory Committees. Exchange of information with ASTM groups is on a less formal basis, than is the case with USASI, and awareness of programs of the many ASTM standards groups depends more upon individual contacts.

Perhaps no other area of science and technology covers such a wide scope as the relatively new and rapidly growing field of Nuclear Technology.

This Symposium provided a forum for learning of the many organizations, both national and international, which meet and interact, and whose standardization activities are within the area of nuclear chemistry and technology. This is an area which has been the scene of major scientific and technical research and engineering accomplishment, and one which promises to provide a major source of our energy requirements.

In planning the Symposium and reproducing, on an informal basis, many of the talks which were given, it is our sincere hope that participants and audience alike will view the meeting and this report as an opportunity to tell others about their own involvement in nuclear standards problems, also, to learn of the many avenues for standardization being explored in the field of nuclear chemistry and technology, and finally, to learn of the unfulfilled needs and future demands for standards in this area.

The Symposium made it clear that many important activities are in progress in this field and that additional and continuing communication and effort is needed.



## THE NUCLEAR STANDARDS BOARD OF THE USA STANDARDS INSTITUTE (USASI) -- AN OVERVIEW J. W. Landis

The generosity of your chairman, Dr. Wayne Meinke, in granting me 20 minutes of prime time at this symposium to talk about my favorite subject -- the USASI Nuclear Standards Board -- is very much appreciated. I shall endeavor to give you the comprehensive overview that he requested without exceeding my time allotment.

First, a few words about USASI -- the United States of America Standards Institute. USASI is a private non-profit federation composed of more than 100 technical, professional and trade organizations and approximately 1,000 companies. An outgrowth of the old American Standards Association, it was established in 1965 to act as the national coordinating institution through which interested organizations, including Government agencies, may cooperate in establishing, recognizing and improving voluntary technical standards in the United States.

Before launching into the organization and operation of Nuclear Standards Board, a brief summary of the organization and operation of standards boards in general might be helpful.

USASI sets up standards boards in all areas where specialization or the number of existing or scheduled standards on one subject or a group of related subjects justifies such action. Boards can be created only by a two-thirds vote of the Member Body Council.

The Executive Standards Board recommends the initial membership (both organizations and individuals) of each board for approval by the Member Body Council. Individual board members must either represent Member Bodies or other organizations having a substantial interest in the scope of the board or possess special competence in the area of the board's activities.

#### USASI - Overview

According to the "USASI Operating Procedures," a standards board shall:

- (1) Foster, maintain and coordinate standardization projects within its scope; assure that adequate progress is maintained at all times and act to harmonize any conflicts in the standards-making activities under its jurisdiction.
- (2) Review the standards needs of society and initiate new USA standards activity as required to round out the USA standards coverage in the operational field encompassed by the scope assigned to the standards board in conformance with established procedure.
- (3) Make any appropriate recommendations to the Executive Standards Board for the approval of the latter and the Member Body Council on the scope of the board's responsibilities and on any other subject to improve the effectiveness of the board in its standardization work.
- (4) Reorganize the program of work and disband USA Standards Committees operating under the board's jurisliction and report these actions to the Executive Standards Board.
- (5) Approve the scope of each project to be handled by a USA Standards Committee or other group operating within the jurisdiction of the board to assure clarity of purpose and to avoid duplication of effort. Each scope statement shall be reported to the Executive Standards Board.
- (6) Act upon each standard submitted for approval as a USA Standard within its jurisdiction.
- (7) Review, on behalf of the Institute, recommendations for coordinating international standardization activities on subjects falling within the jurisdiction of the board, making appropriate re-

#### Landis

commendations for United States of America participation in the International Activities Committee.

- (8) Conduct conferences or symposia to discuss standardization matters within its scope as required.
- (9) Review periodically all standards under its jurisdiction. Those standards which have not been reaffirmed or revised within a five (5) year period shall be called to the attention of the sponsors with a request that such bodies take necessary steps for bringing these standards to a current status.

The Nuclear Standards Board consists of 65 members and alternates, representing 33 organizations. These organizations are:

American Chemical Society

American Federation of Labor-Congress of Industrial Organizations

American Industrial Hygiene Association

American Institute of Chemical Engineers

American Insurance Association

American Mutual Insurance Alliance

American Nuclear Society

American Public Health Association

American Society of Civil Engineers

American Society of Mechanical Engineers

American Society of Safety Engineers

American Society for Testing and Materials

Atomic Industrial Forum

Bureau of Explosives

Electric Light & Power Group

Electronic Industries Association

Health Physics Society

Institute of Electrical and Electronics Engineers International Association of Governmental Labor Offi-

cials

## USASI - Overview

Manufacturers Standardization Society of the Valve and Fittings Industry Manufacturing Chemists' Association National Bureau of Standards National Council on Radiation Protection & Measurements National Electrical Manufacturers Association National Fire Protection Association National Safety Council Scientific Apparatus Makers Association Society of Automotive Engineers Underwriters' Laboratories U. S. Atomic Energy Commission U. S. Department of Health, Education and Welfare U. S. Department of Labor, Bureau of Labor Standards U. S. Department of Transportation, Office of Hazardous Materials

The Nuclear Standards Board meets as often as required each year -- usually twice -- and conducts much business by correspondence and letter ballot. Its primary responsibilities are: (1) to insure that nuclear standards are prepared where and when they are required, (2) to insure that nuclear standards are written by knowledgeable people, and (3) to organize all nuclear standards work so that approved standards represent a consensus of all interested parties.

Continuity of administration and policy-making is provided by the Executive Committee of the Board, consisting of seven regular members and one ex officio member (chairman of COINS -- Committee on International Nuclear Standards):

J. W. Landis -- Gulf General Atomic Incorporated

R. E. Kettner -- Nuclear Assurance Corporation

R. G. Chalker -- North American Rockwell Corporation

W. F. Witzig -- Pennsylvania State University

E. C. Barnes -- Westinghouse Electric Corporation

D. C. Fleckenstein -- General Electric Corporation

#### Landis

- G. M. Kavanagh -- U. S. Atomic Energy Commission Alternate -- C. K. Beck
- J. H. Bach -- Westinghouse Electric Corporation (Ex officio)

The Executive Committee meets from 10 to 12 times a year. Among many other duties each member is responsible for maintaining liaison with one or two of the 12 standards committees operating under the aegis of the Board. Normally, contacts with the Executive Committee by the various standards committees are made through these channels.

The main work of the Board is performed by the standards committees to which I have referred. These committees are presently developing 135 subjects, most of which should culminate in specific standards. Each committee is sponsored by one of the organizations active in the nuclear field, which organization often has committees of its own actually writing standards.

The best way to give you a clear picture of the activities of the Board is to summarize the activities of these committees. This may prove to be a rather boring exposition for some of you. With your indulgence, however, I shall proceed because this discussion is essential to your understanding of the current status of nuclear standards work.

Standards Committee Nll, Basic Materials and Testing of Materials Involved in Nuclear Applications, was organized in May 1967 under the sponsorship of the American Society for Testing and Materials (ASTM).

The scope of this Committee is as follows:

Standards for the specification of chemical composition and physical and mechanical properties of materials used in or resulting from nuclear applications and methods of testing and analyses of these materials, but not to include the specification and testing of components made from materials.

The chairman is W. R. Smith, Sr., of General Electric.

The Committee is composed of approximately 25 members with representation from producers, consumers, general interest and government agencies. In addition to the regular membership there are approximately 20 consulting members who maintain liaison with the various ASTM committees.

Seven standards are being developed by the Committee specifically for the nuclear field. In addition, hundreds of other standards are being developed and/or modified in cooperation with ASTM which will be used by the nuclear industry.

The approved scope of Standards Committee N12, Nuclear Terminology, Units, Symbols, Identification and Signals, is:

Standards for nomenclature, definitions and units; identification means such as symbols, signs, labels or color codes; and warning means or devices for all nuclear and radiation activities.

This Committee has completed three standards -- "Glossary of Terms in Nuclear Science and Technology," "Immediate Evacuation Signal" and "American Standard Radiation Symbol" -- and is developing four additional standards. One of the latter -- "Administrative Practices in Radiation Monitoring" -- is in the final stage of preparation.

The chairman of N12 is E. C. Barnes of Westinghouse; the sponsor is the Atomic Industrial Forum.

N13, Radiation Protection, is one of the most active committees in the nuclear field. It is working on 16 standards, six of which have been assigned an "urgent" priority and should be completed this year:

(1)	Uranium	Mines	and	Mills	 Atmospheric Sampling
(2)	Uranium	Mines	and	Mills	 Sampling Patterns
(3)	Uranium	Mines	and	Mills	 Record Keeping
(4)	Uranium	Mines	and	Mills	 Atmosphere Control
(5)	Uranium	Mines	and	Mills	 Direct Measure of Ex-
	posure	e			

(6) Photographic Dosimeters

#### Landis

The scope of N13 is quite broad and will keep this committee in a frenzy of activity, I'm afraid, for many years:

Standards that have general applicability or are related to the protection of individuals and to environmental contamination from radiation or radioactive material.

The chairman of N13 is J. W. Healy of Los Alamos Scientific Laboratory and the sponsor is the Atomic Industrial Forum.

Standards Committee N14, Transportation and Packaging of Fissile and Radioactive Materials, comprising about 40 members, is divided into seven subcommittees dealing with the following subjects:

- Small source fissile materials not calling for special shielding.
- (2) Large sources (fissile and non-fissile materials and irradiated fuel) in Type B packages.
- (3) Waste and other low specific activity materials, including bulk materials.
- (4) Small source radioactive materials, excluding fissile materials, neutron sources and radioisotope devices.
- (5) Transport of radioactive materials through tunnels and over bridges and turnpikes.
- (6) Radioisotope devices.
- (7) Compendium of tests.

The scope of this Committee, which I should have given prior to the subcommittee breakdown, is:

Standards for the packaging and transportation of fissile and radioactive materials but not including movement or handling during processing and manufacturing operations.

The chairman is R. T. Waite of the American Insurance Association.

This Committee is developing eleven standards at present, six on an urgent, or as-soon-as-possible, basis. It is operating under the sponsorship of the American Insurance Association.

Twenty-three people, representing 15 different organizations, are serving on Standards Committee N15, Methods of Nuclear Material Control, which is headed by R. L. Delnay of Dow Chemical. Just recently organized, this Committee has identified twenty areas in which standards activity should be initiated, but has not had time to carry the work to the point of actual writing.

Seven subcommittees have been formed to handle this activity, chairmen have been selected and liaison has been established with ASTM, the organization to which the Committee's scope is most closely related -- except of course for the sponsor, the Institute of Nuclear Materials Management.

The scope of the Committee is:

Standards for the accountability of fissionable materials in all phases of the nuclear fuel cycle, including analytical procedures where necessary and special to this purpose.

Standards Committee N16, Nuclear Criticality Safety, is sponsored by the American Nuclear Society. There are nine organization and two individual members; the Committee roster and the scope are being voted on right now by the Nuclear Standards Board.

An organization meeting was held on January 22, 1968, at which the following scope was proposed:

> Standards for determining the potential for nuclear criticality of fissile materials outside reactors, for the prevention of accidental criticality, and for coping with accidents should they occur.

Since Subcommittee 8 of the ANS Standards Committee has essentially the same scope, N16 intends to depend on the work of this Subcommittee for its input rather than establish its own standards-writing subcommittees, at least for the immediate future. This Subcommittee has referred three proposed standards to N16 for consideration:

"Nuclear Criticality Safety Standard for Operations with Fissionable Materials Outside Reactors," Revision of N6.1-1964

"Standard for Safety in Conducting Subcritical Neutron-Multiplication Measurements in Situ," ANS 8.4

"Criticality Accident Alarm System," ANS 8.3

Copies of these standards are being sent to the members of N16 for information and they will be voted on at the Committee's next meeting.

The chairman of Nl6 is A. D. Callihan of Oak Ridge National Laboratory.

The current work of N18, Nuclear Design Criteria, one of the most important Standards Committees in the nuclear area, is concentrated on five high-priority projects:

- (1) Guide to Application of Single Failure Criterion
- (2) Safety Criteria for the Design of Station PWR Plants
- (3) Class I Electrical Power Systems
- (4) Proposed Criteria for Nuclear Power Plant Protection Systems for Trial Use
- (5) Testing of Containment Vessels

Seven other standards are being developed and the Committee is maintaining a close relationship with N42 and N45. The approved scope is simply:

Nuclear aspects of design criteria for nuclear facilities.

The chairman is W. A. Chittenden of Sargent & Lundy and the sponsor is the American Nuclear Society.

N42, Nuclear Instruments, is concerned with standards for instrumentation in the nuclear field. This covers a broad spectrum from laboratory instrumentation for radiation research to health physics and prospecting instruments to monitoring and control systems for nuclear reactors. The Committee also serves as the U. S. advisory group in connection with the work of Committee TC45 of the International Electrotechnical Commission and its subcommittees. In this connection, N42 personnel review the numerous international standards proposals of TC45 and provide comments on the basis of which a U. S. position is formulated.

Standards have been issued on bases for GM counter tubes and quartz-fiber electrometer type chargers, and definitions for the scintillation counter field were approved and included in the USASI nuclear glossary recently published. A standard on connectors for nuclear instruments has recently been approved and referred to the Nuclear Standards Board. N42 is also involved in a number of standards on nuclear reactor control systems generated by IEEE committees (IEEE is the sponsor of N42). IEEE Detector Committee drafts on test procedures for semiconductor radiation detectors and for amplifiers and preamplifiers for semiconductor radiation detectors have been reviewed by N42 and the Committee is therefore in a position to take rapid action on these standards when they are referred to the Nuclear Standards Board.

The membership of N42 is essentially the same as that of N3, which it supersedes, though its scope has been broadened somewhat to include mechanical, hydraulic and pneumatic instrumentation, in addition to the electrical and electronics instrumentation with which N3 was concerned. Twenty organizations are represented on N42. The representatives, including six alternates and four members-at-large, total 30.

The chairman of N42 is Louis Costrell of the National Bureau of Standards.

Standards Committee N43, Equipment for Non-Medical Radiation Applications, is making exceptionally good progress. It has 48 members and alternates, from 28 different organizations. It has already produced a standard entitled "Sealed Sources Classification Guide" and is well along with the preparation of five other standards.

Its sponsor is the National Bureau of Standards and it is chaired by L. H. Horn of Underwriters' Laboratories. The approved scope reads as follows:

Standards for the preparation and use of radioactive materials and radiation sources for scientific, industrial and educational purposes.

Subcommittees have been formed to handle the bulk of the Committee's work. These subcommittees operate in the following areas:

- (1) Diffraction and fluoroscopic analysis equipment.
- (2) Self-luminous products.
- (3) Sealed sources and devices.
- (4) Particle accelerators.
- (5) Review of NBS Handbook #93.
- (6) Review of NBS Handbook #66.

Standards Committee N44, Equipment and Materials for Medical Radiation Applications, is just being organized by its chairman, R. T. Moore, and its secretary, L. R. Setter, both associated with the National Center of Radiological Health of the U. S. Public Health Service, the sponsor. The first official organizational meeting will be held on September 24, 1968. There is nothing else to report at this time except that the scope established by the Nuclear Standards Board reads as follows:

Standards for the preparation and use of radioactive materials and radiation sources in medical applications.

N45, Reactor Plants and Their Maintenance, under the leadership of W. M. Joslin of Commonwealth Edison, probably has more ground to cover than any other Standards Committee. The fifteen standards being developed range from "Requirements and Rules for Examination and Inspection of Pressure Retaining Equipment of Nuclear Power Reactors" to "Standard Guide for Cleaning Nuclear Reactor System Components." One standard, "Safety Standards for Design, Fabrication, and Maintenance of Steel Containment Structures for Stationary Nuclear Power Reactors," has already been published. Excellent progress is being made and the Committee has pioneered the formation of unofficial "steering groups" with other Committees so that overlap, wasted effort, etc., are held to a minimum.

Three technical societies have demonstrated active interest in this Committee. They are the American Nuclear Society, the Institute of Electrical and Electronics Engineers and the American Society of Mechanical Engineers. ASME is the official sponsor.

The scope of the Committee is:

Standards for the location, design, construction and maintenance of nuclear reactors and plants embodying nuclear reactors, including equipment, methods and components special for this purpose.

Three task forces have been set up to tackle the highpriority problems that have arisen in the N45 area of responsibility:

In-Service Inspection Task Force

Chairman -- W. P. Johnson

Construction-Phase Quality Assurance Task Force

Chairman -- B. F. Langer

Reactor Containment Task Force

Chairman -- R. N. Bergstrom

The final Standards Committee -- N101, Nuclear Fuel Cycle Facility, Location, Design, Construction and Operating Criteria -- is not quite a catch-all, but almost. Its responsibilities, as indicated by its scope, "Standards for the location, design, construction and operation of atomic facilities except nuclear reactor facilities," are variegated and somewhat indefinite in certain areas. The Nuclear Standards Board is attempting to ameliorate this situation.

Sponsored by the American Institute of Chemical Engineers, and chaired by M. M. Braidech (retired), this Committee is in the process of reorganization, but has pinpointed 17 standards that should be written and has started work on nine of these.

As I have indicated, these committees carry most of the nuclear standards workload. This means that chairmen, to be successful, must be talented, knowledgeable and hardworking. They are -- and I salute them for their leadership, dedication, determination and patience. I cannot pass up this opportunity, however, to remind them once again that they have six important duties:

- To formulate comprehensive lists of the standards that should be prepared.
- (2) To assign priorities and schedules to these standards.
- (3) To set up task forces to tackle the urgently needed standards.
- (4) To set up subcommittees to prepare all other standards.
- (5) To see that satisfactory progress is made on all fronts.
- (6) To report the status of their work to the Executive Committee as required.

The message I want to leave you with -- and I hope that the information I have given you today substantiates this -is that the Nuclear Standards Board is fully aware of the extreme importance of a comprehensive and well-thought-out system of standards to the success of the nuclear industry and is moving aggressively to provide such a system. Pressure has been put on each chairman to accelerate his committee's activities and arrangements are being made with many industrial companies, government agencies and other organizations to provide top-flight personnel to do the massive amount of work involved. We are receiving splendid cooperation from everyone -- particularly from the U. S. Atomic Energy Commission and the U. S. Public Health Service. With help like that being provided by these two key agencies, we believe that we shall meet all of our objectives.



## THE INTERNATIONAL STANDARDS ORGANIZATION (ISO) ACTIVITIES OF TC85 COMMITTEES D. C. Fleckenstein

Earlier speakers have described national programs for standardization in the nuclear field. While subsequent papers will also discuss international activities, this paper is to extend the previous discussion of the United States of America Standards Institute to the international level. The other papers will describe international organizations which are essentially independent of USASI.

Although this paper is concerned with the ISO and, in particular, activities of the TC85 committee of that organization, a few general remarks about international standardization will be useful background for the "hows", "whys" and "whats" of this activity.

At the risk of being repetitious, standards are based on the consolidated results of science, technology and experience. The evolution of any standard requires a scientific approach, an advanced level of technology and a breadth of knowledge and experience. Clearly it is all to the good if these can be gathered together in a world forum.

The necessity for international standardization is becoming increasingly urgent in the light of the rapid advancement of technology and the growth of world trade. It has been said that it is always too soon to fix a standard and always too late: too soon because subsequent experience or technique may supersede a decision made now: too late because existing practice has diverged from the standard about to be set up.

International standardization is particularly desirable before different countries adopt irreconcilable solutions. It is much more difficult to correct the chaos of the past than to keep standards abreast or even ahead of current practice. Failure to reach international agreement at the right

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time may mean that some opportunity has been lost for a long time. An internationally recommended standard must be capable of wide application and it is more capable of this if many countries are able to conform to it.

With this brief statement regarding "why", I'll turn to the "how" of international nuclear standardization through USASI. USASI has membership in two international organizations: the ISO which is the International Organization for Standardization and the IEC, the International Electrotechnical Commission. The latter organization is deserving of mention at this time because some of the work of ISO TC85 is related to activities within the IEC.

The International Electrotechnical Commission was founded in 1906, its first president being Lord Kelvin. The Commission's work embraces almost every sphere of electrotechnology, from power, which was its earliest concern, to telecommunications, electronics and electro-domestic appliances. In addition to promoting the standardization of materials and equipment, the commission aims to improve understanding between electrical engineers of all countries by drawing up common means of expression.

The International Organization for Standardization to which the IEC is affiliated, is a younger body. It was founded in 1947 to carry on the work of the International Federation of National Standardizing Associations which had been set up in 1926 but whose work was interrupted by the second world war. The object of ISO is "to promote the development of standards in the world with a view to facilitating the international exchange of goods and services and to developing mutual cooperation in intellectual, scientific, technological and economic activity."

The ISO with 55 member bodies and the IEC, with 40, represent countries having four-fifths of the world's population. Between them the two organizations have published nearly 1,000 recommended standards and there are about 1,000 recommendations currently in draft.

#### Fleckenstein

It must be recognized that the ISO and IEC cannot impose international standards. That is why their publications are termed Recommendations. The results of their work must be acceptable for their inherent value without resort to authoritarian or compulsory measures. The member organizations have, however, a moral obligation to use the Recommendations as the basis for their own national standards so far as local conditions will permit.

To meet its previously stated objective the ISO may:

- Take action to facilitate coordination and unification of national standards and issue necessary Recommendations to member bodies for this purpose;
- b) Set up international standards provided, in each case, no member body dissents;
- c) Encourage and facilitate, as occasion demands, the development of new standards having common requirements for use in the national or international sphere;
- Arrange for exchange of information regarding work of its member bodies and of its technical committees; and
- e) Cooperate with other international organizations interested in related matters, particularly by undertaking at their request, studies relating to standardization projects.

The 55 ISO members are the national bodies most representative of standardization (one for each country) who have agreed to abide by the organization's constitution and rules of procedures. Organizations in developing countries which do not have a standards body may become correspondent members. Countries represented by correspondent members cannot be accepted as member bodies until they have established their own national standards body.

Technical work of the ISO is carried out by its technical committees. These committees are composed of a delegation from each of the member bodies wishing to take part in

### ISO-TC85

the work. Each technical committee has a secretariat, which is undertaken by a member body designated by the Council, the administrative organ of the ISO. In its capacity as secretariat the member body acts impartially: it has its own delegation with exactly the same status as other participating members of the technical committee. The secretariat is responsible for the satisfactory conduct of the work of the technical committee and has to report annually to the Council on the results achieved.

Technical committees may agree to establish subcommittees and/or working groups as necessitated by the work program. Subcommittees must comprise at least five member bodies and one of the member bodies is elected to serve as the secretariat.

The 1967 ISO <u>Memento</u> lists 116 technical committees and 149 subcommittees. As a point of interest, the United States holds the secretariat for 10 of the technical committees including Technical Committee 85, "Nuclear Energy".

TC85 with a scope of "Standardization in the Field of Nuclear Energy and its Peaceful Applications", first met in 1957. At its first meeting the 23 participating member bodies established three subcommittees:

Subcommittee 1, "Terminology, Definitions, Units and Symbols", with the USA as secretariat, is concerned with the development of Recommendations for technical units, terminology, definitions and symbols in all fields of nuclear energy.

Subcommittee 2, "Radiation Protection", with the secretariat in France, is to prepare Recommendations relating to radiation protection and the associated methods of measurements, both for workers and the general public.

Subcommittee 3, "Reactor Safety", with the secretariat assigned to the United Kingdom, was established to prepare Recommendations on those aspects of the administration, design and construction and operation of reactors which have a bearing on safety.

### Fleckenstein

At its second meeting in 1958 TC85 established Subcommittee 4, "Radioisotopes", with Poland as the secretariat. SC4 is to consider all aspects concerned with the production, measurement, handling and all forms of use of radioisotopes, natural and artificial.

TC85 met next in 1960 and most recently in November of 1967. At the time of the November meeting the secretariat reported that although TC85 had not met since 1960 there had been more than 25 meetings of subcommittees and working groups held in the interim period. So far, continued the secretariat's report, TC85 had published just one ISO Recommendation. Such a production rate leaves something to be desired so that a main item of discussion at the November meeting was the exploration of methods to speed up the work. Some of these methods are being tried now.

There is a lot of work underway and a number of working groups were established by each of the subcommittees. Because working groups are responsible only for the study of a particular question, some have come and gone while still others persist. For the purpose of this review it's unnecessary to detail the work of the various subgroups of the subcommittees of TC85. The listing of the 4 subcommittees and their respective fields of interest reveal the current direction and activity in nuclear energy standardization at the ISO level.

As noted earlier the USASI is a member of ISO and holds the secretariat for TC85. Participants in the USASI nuclear energy standardizing programs have, therefore a direct connection with like international programs. The secretariat of TC85 resides in the Nuclear Standards Board of USASI. Further, TC85 subcommittee actions are of interest to specific standards committees of the Nuclear Standards Board. For example, TC85/SC2, "Radiation Protection" is closely allied with the USASI N13, "Radiation Protection". Although an ability to use the word "identical" when comparing SC2 with N13 would make the matter of coordination a great deal sim-

### ISO-TC85

pler, such a relationship is not the case. For instance, the broad scope statement of SC2 enabled it to initiate, last November, a fourth working group to consider apparatus for gamma radiography. Under the reorganized Nuclear Standards Board, such equipment falls outside the scope of N13 and into the interest sphere of N43. Therefore, this SC2 activity will be followed by a representative of N43.

Lacking a one-to-one correspondence in organization and a need to provide some direction and motivation to TC85, a responsibility of a secretariat, the Nuclear Standards Board, established in November, 1965 its committee on International Nuclear Standards known as COINS. COINS was charged with the responsibility to act for the Nuclear Standards Board in all ISO nuclear standards activities and was designated as the U.S. agency to advise on United States action in ISO TC85 matters.

With these and other charges COINS is the focal point in USASI for international nuclear standardizing activities. These activities include some 15 TC85 subcommittees and subgroups and eight IEC subcommittees and subgroups which in turn involve eight of the standards committees of the Nuclear Standards Board.

A few examples will illustrate these relationships.

Turning first to the IEC, TCl is entitled "Terminology". Within TCl there are two publications of interest, the first "Nuclear Power Plants for Electric Energy Generation" and the second "Detection and Measurement of Ionizing Radiation by Electric Means." These activities of IEC TCl, therefore, are of specific interest to N12, "Terminology, Units, Symbols, Identification and Warning".

IEC TC45, "Electrical Measuring Instruments Used in Connection With Ionizing Radiation", SC45A, "Reactor Instrumentation" and SC45B "Health Physics Instrumentation" are of interest to:

> N13, "Radiation Protection" N18, "Nuclear Design Criteria"

- N42, "Nuclear Instruments
- N43, "Equipment for Non-Medical Radiation Applications"

N45, "Reactor Plants and Their Maintenance"

Finally from the IEC listing is TC62, "X-Ray Medical Equipment" which, of course, is of interest to N44, "Equipment and Materials for Medical Radiation Applications". It seems quite possible that because one of the subjects to be dealt with by TC62 is X-ray protection in X-ray medical equipment, N13 will also have an interest. TC62 is relatively new, having had its first meeting in the spring of this year, so that programs for it are not as clearly defined as they are for the other committees.

Turning briefly to ISO TC85 the same USASI committees are involved with work of the subcommittees and subgroups of ISO TC85. Additionally, within Subcommittee 4, that SC concerned with all aspects of the production, measurement and handling of radioisotopes, a working group on the packaging and transport of isotopes is within the scope of N14, "Transportation of Fissile and Radioactive Materials".

Despite the fact that only one Recommendation had been approved, TC85 committees have been very active. Again by way of illustration, a brief summary of the report that was made at the conclusion of the meeting last November, will support this view. Some things have changed in the 10 months since that meeting however, those details won't affect this particular discussion.

### Subcommittee 1

Subcommittee 1 approved the "Nuclear Energy Glossary" (Second Edition) and forwarded it for balloting. Subcommittee 2

Subcommittee 2 considered a draft document on air sampling, one on personal photographic dosimeters and two drafts on direct and indirect reading pocket dosimeters. Moreover, SC2 noted that its first draft proposal of "The Fundamental Principles for Protection

### ISO-TC85

in the Design and Construction of Installations for Work on Unsealed Radioactive Materials" had been balloted by TC85 and it was ready for balloting by the member bodies.

Further, SC2 decided to pursue test methods for exposure and rate meters and had established Working Group 4 on Equipment for Gamma Radiography. In the future WG1 would study filter sizes for air sampling, WG2 would attempt to establish reference rays for X-ray and gamma radiation for calibration of photographic dosimeters and WG3 would work on portable dosimeters and rate meters.

## Subcommittee 3

Subcommittee 3 reported that the work of WG2, "Meterorological Aspects", was to be held in abeyance pending the outcome of work by the IAEA; established a study group to survey future work in the field of steel containment structures; WG4 on Irradiation of Steels was disbanded; WG5 on Criticality Safety was disbanded; established a study group in the area of prestressed concrete pressure vessels and prestressed containment structures; and established a study group to consider the possibility of collecting and statistically analyzing information concerning faults relating to reactors and their equipment.

### Subcommittee 4

Subcommittee 4 reported that at its meeting the SC dealt with the revision of the draft proposal on the contents leakage test and radiation leakage test, the program of future work on sealed sources and the results of the inquiry on the possibility of standardization of transport packaging of radioactive sources. Hopefully this summary provides a better understanding

of the "why" "how" and "what" USASI is involved in ISO nuclear standardization. The "why" and "how" are fairly well

established, the "what", however, is subject to change and depends in large part on the interest of those who are willing to participate in satisfying their understanding of the "why".



# THE INTERNATIONAL COMMISSION ON RADIOLOGICAL UNITS AND MEASUREMENTS (ICRU): AN OVERVIEW WITH SPECIAL EMPHASIS ON RADIOACTIVITY MEASUREMENTS W. B. Mann

I note from the title of my paper that I am to give an overview of the activities of the International Commission on Radiological Units and Measurements. The word "overview" bothers me a little because I feel that it must be the opposite of "worms-eye view", but I shall try to rise to the occasion.

The International Commission on Radiological Units and Measurements (ICRU) and the International Commission of Radiological Protection (ICRP) both report to the International Congresses Radiology.

The ICRU was formed in 1925 under the auspices of the first International Congress of Radiology in London, and started then as the International X-Ray Unit Committee.

At the Second International Congress of Radiology in 1928, held in Stockholm, the <u>roentgen</u> was defined and certain recommendations adopted relating to X-radiation protection. The International Congresses of Radiology were later adopted by the International Society of Radiology which thereby became the parent, or grand-parent, organization of both ICRU and ICRP.

There have been, through the years, only slight changes in the name of the ICRU. In 1931, the International X-Ray Committee became the International Committee for Radiological Units and then, at the 1950 International Congress of Radiology in London, became a Commission. In 1953 it became the International Commission on Radiological Units and Measurements and that year, in Copenhagen, defined the <u>rad</u> as the unit of absorbed dose.

## ICRU - Overview

Through all these years, since 1928, the chief architect of the ICRU has been Lauriston S. Taylor in his capacity as member, secretary and chairman.

The ICRU is composed of some 10 to 12 members from various countries together with a Chairman, Vice-Chairman and Secretary (see last section of the Appendix to this paper).

The ICRU has consultative status with a wide variety of international organizations which are enumerated in the fifth section of the Appendix to this paper. In particular it is recognized by the World Health Organization as its body of technical advisors in the field of radiological units and measurements. The ICRU also has consultative status with the United Nations International Atomic Energy Agency (IAEA) and in 1959 co-sponsored with the IAEA the first Symposium on Radioactive Metrology, held in Vienna in 1959, in connection with which I had the honor of representing ICRU and collaborating with Professor A. Sanielevici in its organization.

The functions of the ICRU may perhaps best be described by reading the headings from its position plan given in the 1959 ICRU Report in NBS Handbook 78 (page 83). These are:

- I. Continued study and development of definitions and basic concepts in the general field of units and measurements of ionizing radiation
- II. At some international focal point, to keep informed on and evaluate the measurement standards and the intercomparison of such standards required in the radiation field
- III. To study and make recommendations relative to the practical measurements of radiation
  - IV. To continue the development of the principles and practices of radiation dosimetry
    - V. Primarily for medical radiology, to establish some guidance and international agreement on the methods of testing the basic characteristics of certain essential component radiological equipment.

VI. To provide a collaborative and working relationship at some central focal point between various international organizations having special needs or interests in the field of radiation units, standards, and measurements.

Currently the ICRU attempts to achieve these functions through a system of Planning Boards and Task Groups that are enumerated in the third section of the Appendix to this paper. Of most immediate interest to the members of the American Chemical Society here today is the program of Planning Board 1A and its three Task Groups which work under the respective chairmanships of Dr. R. Dudley, Mr. S. B. Garfinkel and Dr. R. G. Wood. Task Group No. 2 has recently issued its report, ICRU Report 12 on the Certification of Standardized Radioactive Sources by S. B. Garfinkel and G. R. Newbery.

The ICRU has long sponsored international comparative measurements of samples of radionuclides. Some intercomparative measurements in radioactivity had already been undertaken independently of ICRU in the late 1940's, by the laboratories of Canada, the United Kingdom and the United States. These measurements were greatly widened in scope, however, in the 1950's, under the aegis of the ICRU, and they are now sponsored by the Bureau International des Poids et Mesures (B.I.P.M.). In 1953 the ICRU had set up a Subcommittee on Standards of Radioactivity to extend the intercomparisons to countries other than the initial three. This Subcommittee was in subsequent years replaced first by its Committee I on Standards and Measurements of Radioactivity for Radiological Use and now by its Planning Boards IA and IB.

The results of the ICRU sponsored intercomparisons are summarized in fair detail in the 1959 and 1962 ICRU Reports (NBS Handbooks 78 and 86).

As an example of such intercomparisons organized internationally by the ICRU, I might briefly quote that of radium as a sort of case history. As an introduction I would, how-

ever, like to show two historic documents (figure 1), namely the certificates of the United States 1913 and 1936 Primary Radium Standards. The earlier standard had been brought to this country by Mme. Marie Curie and the later one was one of the twenty standards prepared by Professor O. Hönigschmid from material used in his determination of the atomic weight of radium, and sealed by him on the afternoon of June 2, 1934.

There is a striking permanence of the International Radium Commission in the persons whose signatures appear on the certificates over a period of nearly a quarter of a century. Stefan Meyer is the same; Mme. Curie has been succeeded by her daughter Irene Curie; and Ernest Rutherford is the same, apart from age, but has lost in the meantime his first name, which is one of the disadvantages associated with becoming a peer of the British realm!

The first intercomparisons of Hönigschmid radium standards were directly arranged between the U.K. National Physical Laboratory (NPL) and the U.S. National Bureau of Standards (NBS) in 1954. These were followed by many others, the intercomparisons being stimulated to some extent by the construction at the NBS of a precision microcalorimeter with dimensions specially chosen to accommodate the Hönigschmid standards. Prior to this all intercomparisons of these standards had been based on gamma-ray measurements, many by means of gold-leaf electroscopes, and many of them were relative to the Paris and Vienna International Radium Standards.

The great wealth of data that began to be accumulated on the Hönigschmid standards needed some forum for international discussion, coordination and analysis. This forum was provided by the ICRU Subcommittee on Radioactivity Standards and Committee I, and the results obtained prior to that date were discussed in the 1959 ICRU Report (NBS Handbook 78). In this report there is a statistical analysis of the data, carried out by W. S. Connor and W. J. Youden who also derived "best estimates" for the values of the standards, using both Hönigschmid's weighings and the more recent experimental data.

# COMMISSION INTERNATIONALE DES ÉTALONS DE RADIUM.

# CERTIFICAT.

Das als Chlorid dargestellte Radiumpräparat Nr... 6. entstammt St. Joachimstaler Uranpechblende und ist demnach praktisch frei von Mesothor.

Es enthält 21 50 Milligramm Salz.

Es wurde am *1. Juli 1913* eingeschlossen in ein Glasröhrchen (Thüringer Glas) von 027 mm Wandstärke, äußerem Durchmesser 32 mm, Länge 42. mm, an dessen Ende ein feiner Platindraht eingeschmolzen ist.

Dasselbe wurde als Secundärer Standard an den Wiener Etalons und an dem internationalen Standard in Paris nach mehreren T-Strahlungsmethoden unabhängig voneinander geeicht.

Der Y-Strahlung nach ist es im Jahre 1913 äquivalent 20-28 mg RaCl<sub>4</sub>. (Die jährliche Abnahme beträgt etwa 0.4 Promille.)

Unter Zugrundelegung der Atomgewichte von

	26 35:457 79:916	für	Radium Chlor Brom
ht i	dies		

15.44 mg Ra-Element, 20.28 mg RaCl., 26.36 mg RaBr.

entsprid

Die Genauigkeit dieser Angabe wird auf **0.2** % für gesichert gehalten.

Fin die Wiener Messung

Stefan Meyer

La Préparation de Chlorure de Radium contenue dans l'ampoule Nr. & provient de la pechblende de St. Joachimsthal. Elle est donc pratiquement exempte de Mésothorium. Elle contient & GD. Milligrammes de sel.

Le sel a été enfermé le 1/17 1913 dans un tube de verre (Verre de Thuringe). Epaisseur du verre 0:27 mm; Diamètre extérieur 3:2 mm; Longueur 222 mm. Un fil de platine fin a été soudé à l'extrémité du tube.

En qualité d'Étalon escondaires l'ampoule a été comparée à l'Étalon de Vienne et à l'Étalon International de Paris, au moyen de méthodes de mesures basées sur le rayonnement r. La comparaison a été faite indépendamment à Vienne et à Paris.

D'après son rayonnement 7, la Préparation équivaut en l'année (913 à **20.21** mg. RaCl., (La diminution par année est de 0'4 pour mille.)

		ome				79-916	
uit	la	tene	u	r	co	rrespondan	

on déduit la teneur correspondante en Radium élément et en Bromure de Radium:

Ra	75 77 mg,
	20.28 mg.
RaBr,	26.36 mg.

La précision de ces résultats est considérée comme assurée à une approximation de  $\mathcal{O}\mathcal{I}^{\circ}/_{o}$ .

Four les useunes faites à Paris

Mr. Curres

Specimen No. 6 of Radium is prepared as chloride from pitchblende of St. Joachimstal and is consequently practically free from Mesothorium.

It contains 250 Milligrammes of salt.

It was enclosed the  $1/p^{-}$  19/3 in a glass tube (Thuringian glass) of 0:27 mm thickness, exterior diameter 3:2 mm, length  $1^{40}$  mm, a thin platinum wire being fused into the end of the tube.

It is calibrated as Secondary Standard by comparison with the Vienna-Standard and with the International Standard at Paris, several independent 7-raymethods being used.

Measured by the T-radiation, it is in the year /9/3 equivalent to 20-29 mg. RaCl.. (The yearly decay is about 0'4 per mille.)

Taking the atomic weights

226	for Radium
35.457	for Chlorin
79-916	for Bromin-

this corresponds to

/5'44 mg Ra-element, 20.28 mg RaCl<sub>2</sub>, 26 36 mg RaBr<sub>3</sub>.

These statements are considered correct to  $\partial \cdot \mathbf{Z}^{0}/_{0}$ .

President the Commission

E Rutherful

Figure 1. Certificate United States - 1913

# COMMISSION INTERNATIONALE DES ÉTALONS DE RADIUM.

# CERTIFICAT.

Das als Chlorid dargestellte Radiumpräparat Nr. AJZ entstammt Uranerzen von Katanga, belgisch Kongo, und ist demnach praktisch frei von Mesothor.

trei von Mesothor. Es wurde am 2. Juni 1934 von Prof. Hönigschmid in München aus-gewogen und in ein Glasröhrchen von 3 mm lichter Weite und 0·27 mm Wandstärke und einer Länge von 36 ...m eingebracht, an dessen Ende ein feiner Platindraht eingeschmol-

ein teiner Platindraht eingeschmol-zen ist. Es ist gekennzeichnet durch die kos Schutzrohres, sowie durch sein Bruttogewicht von 323 73 mg und enthielt damals 30-22 mg reines, wasser-freies Salz, gewichtsmäßig entsprechend 32:24 mg Radiumelement. Der Reinheitsgrad des verwen-deten Radiumelhorids ist sichergestellt durch die von Proc Hönieschmid

deren Radiumeniorias ist sichergesteint durch die von Prof. Hönigschmid ausgeführteAtomgewichtsbestimmung, die für Ra zu dem Werte 226 05 führte, sowie durch die von Prof. W. Gerlach durchgeführte spektroskopische Unter-undung wiehen einen Bazimmerhalt

durchgeführte spektroskopische Unter-suchung, welche einen Bariumgehalt von maximal 0'002 – 0'003 Atom-prozent Barium ergab. Das Radiumpräparat wurde zum letztenmal am 25. Mai 1934 durch eine Fällung mit Schwefelwasserstoff von RaD befreit. Das Präparat wurde mit den primären Etalons von 1911 in Wien und Paris nach mehreren 'Sstrahlungs-methoden unabhängig von einander geeicht. geeicht.

Der Y-Strahlung nach ist es inde 1936 äquivalent 3870 mg Ra-Element, Für eine mittlere Lebensdauer des Radiums von 2295 Jahren beträgt der jährliche Abgang 0·436 Promille. Unter Zugrundelegung der Atomgewichte

gewichte 22605 für Radium 35457 für Chlor ortspricht diesbezogen auf die primären Etalons von 1911 für die Jahras-wende 1936/1935

- 38.10 mg Ra-Element, 50.05 mg RaCl<sub>2</sub>, 65.09 mg RaBr<sub>2</sub>.

Für die Wiener Messungen:

Fian Meyer

Figure 1. Certificate United States - 1936

La préparation de Radium No. XIV est un chlorure qui provient de la pechblende du Katanga, Congo Belge, et est, par conséquent, pratique-ment exempt de Mésothorium.

ment exempt de Mésuthorium. Cette préparation a été pesée le 2 Juin 1934 par le Pr. Hönigschmid a Munich et a été introduite dans un tube en verre de 3 mm de diamètre intérieur, de 0°27 mm d'épaisseur des parois et de 3 & mm de longueur, sur l'extrémité duquel un fil fin de ribuines d'et acult platine a été scellé. Elle est identifiée par le No. 5937

du tube protecteur, de même que par son poids brut de 323.93 mg et elle contenait alors 50.22 mg de sel pur

contenait alors <sup>40</sup><sup>-22</sup> mg de sel pur anhydre correspondant au poids de 20:23 mg de Radium élément. Le degré de pureté du chlorure de Radium est mis en évidence par les mesures du poids atomique faites par le Pr. Hönigschmid, qui conduisent pour le Radium au nombre 226:05, ainsi que ner les ressis spectroscopour le Radium au nombre 22605, ainsi que par les essuis spectrosco-piques faits par le Pr. W. Gerlach, qui donnent une teneur en Baryum au maximum de 0'002 – 0'003 pour cent d'atomes de Baryum. La préparation de Radium a été purifiée pour la dernière fois du RaD le 25 Mai 1934 par une précipi-tation par l'hydrozère sulfuré

KaD le 25 Mai 1934 par une précipitation par l'hydrogène sulfuré. La préparation a été comparée en rayons y aux étalons primaires de 1911 par diverses méthodes, indépendamment à Vienne et à Paris. Par son, rayonnement y elle est équivalent ef méthé à 38° /0. mg de Radium clément. Pour une vie moyenne di D-dium.

Pour une vie moyenne du Radium de 2295 ans, la décroissance annuelle est de 0436 pour mille. Prenant pour base le poids

atomique de 226:05 pour le radium de 35:457 pour le chlore de 79:916 pour le byome il vient pour fais 1936, decour 1937

par rapport de 1911 : 38-10 rig Ra-élément, <u>30</u>03 rig RaCls, 6507 rig RaBrg, do don rapport aux étalons primaires

La précision de ces données est exacte à  $\mathcal{Q}_{\cdot}^{\cdot}$   $\mathcal{J}_{\cdot}^{\circ}$ ,  $\mathcal{O}_{0}^{\cdot}$ . Pour les mesures faites à Paris:

I factor time

The Radium-preparation Nr. X/V has been prepared as chloride from uranium ores from Katanga, Belgian Kongo, and is therefore practically free from Mesothorium.

It was weighed on June 2nd, 1934 by Prof. Hönigschmid in Munich and transfered to a glass tube of 3 mm inner diameter, 0.27 mm thickness of wall and 36 mm length, with a thin platinum wire sealed in at one end.

It is characterised by the Nr. 5 737 It is characterised by the IVT.2.224 on the protection tube, as well as the gross weight of 323:23 mg and contained at the date given above D2.22mg of pure salt free from water corresponding in weight to 38.23 mg redumalement radium-element.

Tadum-element. The degree of purity of the radium-chloride used is warranted by the determination of the atomic weight carried out by Prof. Hönigschmid, which gave for radium the value 226r05, and the spectroscopic investigation by Prof. W. Gerlach, which showed that the barium-content was 0r002 — 0000 per cent atoms at most. 0.003 per cent atoms at most.

RadiumD was separated from the radium-preparation for the last time on May 25<sup>th</sup>, 1934, by precipi-tation with hydrogen sulphide.

The preparation has been compared with the primary standards of 1911 in Vienna and Paris inde-pendently by several <sup>7</sup>-ray-methods.

According to its V-rays it was broard he and 436 equivalent to 38.10 mg Radium-element. For an average life of radium of 2295 years the loss per year is 0.436 per mille.

Taking the atomic weights:

22605 for radium 35457 for chlorine 79916 for bromine as a basis this corresponds for the end of 1936 and he beginning of 1937 compared with the primary standards of 1911 to:

38.10 mg Ra-element, 50.05 mg RaCl2. 65.07 mg RaBr2.

These statements are considered correct to 0.3 °/<sub>0</sub>.

The President :

Rutherford

A summary of the results that had been obtained in various laboratories is shown in Table 1 in which A and D are the U. S. standards, B the British, C the Canadian, G the German, P the Paris and V the Vienna standard. A leastsquares treatment of the ratios shown in column 4 of Table 1 gave experimental ratios which are compared with the weight ratios of Hönigschmid in Table 2. Giving equal weight to both the Hönigschmid and the new experimental ratios of Table 2 and using the method of Connor and Youden (Journal of Research NBS <u>53</u>, 273, 1954), a set of "best estimates" was obtained for the weights of seven Hönigschmid radium standards. These weights, in milligrams, are shown in Table 3.

In 1959 A. V. Astin, the Chairman of the BIPM Consultative Committee on Standards of Measurement for Ionizing Radiations, invited me to act as chairman of an <u>ad hoc</u> Study Group to make recommendations "concerning the future status of the International Radium Standard (Hönigschmid - 1934)". Reviewing the results given in the 1959 ICRU Report the <u>ad</u> <u>hoc</u> Study Group concluded that the normalized system of "best estimates" given in this report could be the basis for a normalized system of international radium standards against any of which relative measurement of radium samples could be made with equal validity. Meeting on July 17, 1959, on the occasion of the Ninth International Congress of Radiology in the city of Munich, where 25 years earlier Hönigschmid had prepared the twenty radium standards that bear his name, the <u>ad hoc</u> Study Group recommended that:

"a normalized system of Hönigschmid 1934 radium standards be considered as the basis of reference for all relative radium standardization and calibration. For all practical purposes at the present time this would be achieved by taking Hönigschmid's masses for those standards currently in use as national or international standards.

Place		and- ards	Ratios	Hönig- schmid Ratios
NBS.       .       .         NBS.       .       .	Calorimeter Electroscope Calorimeter Electroscope Calorimeter Electroscope Calorimeter Calorimeter Electroscope GM counter Electrometer Electroscope GM counter Electroscope Calorimeter Electroscope Calorimeter Electroscope Calorimeter Electroscope Calorimeter Electroscope Calorimeter Electroscope Calorimeter Electroscope Calorimeter Ionization chamber Ionization chamber	A/B A/D A/D A/D A/D A/D A/D D/B D/B D/B D/B D/B D/B D/B D/B D/B D	2.441 2.450 1.870 1.870 1.870 1.869 1.870 1.875 1.875 1.874 1.305 1.308 1.305 1.308 1.304 2.608 2.612 1.395 1.398 1.578 1.583 1.185 1.185 1.185 1.185 1.6527 1.6516	2.450 2.450 1.870 1.870 1.870 1.870 1.870 1.310 1.583 1.181 1.181 1.6535 1.6535
Radiumforschung	Radon ionization.	C/V C/P	1.030 <sub>6</sub> 1.4257	1.0319 1.4274
NPL	chamber Electroscope Electrometer Scintillation counter	C/B C/B C/B	1.546 1.545 1.547	1.5478 1.5478 1.5478

Table	l.	Summary	of	results	of	measurements	on
				d standaı			

Table 2. Comparison of weighed and measured values.

Standards	Experimental	Hönigschmid	Percentage
	ratios	ratios	difference
A/B A/D D/B A/G D/G A/C C/D C/D C/C C/P C/B	2.444 1.872 1.306 2.612 1.395 1.581 1.184 1.652 1.030 1.425 1.546	2.450 1.870 1.310 2.617 1.400 1.583 1.181 1.654 1.032 1.427 1.548	0.24 .11 .31 .36 .13 .25 .12 .19 .14 .13

TABLE 3

Best estimate			
	of radium	Difference	
in milligrams	(June, 1934)	in milligrams	
	in milligrams		
38,208	38 227	-0.019	
15.615		.010	
24.159	24.153	.006	
20.425	20.447	022	
14.614	14.607	.007	
16.935	16.921	.014	
23.428	23.407	.021	
	38.208 15.615 24.159 20.425 14.614 16.935	in milligrams 38.208 38.227 15.615 15.605 (15.604 <sub>6</sub> ) 24.159 24.153 20.425 20.447 14.614 14.607 16.935 16.921	

All national secondary standards have been calibrated in terms of the 1911 or 1934 Paris and Vienna Radium Standards. The 1934 Paris and Vienna Standards have also been most carefully compared with their 1911 predecessors. Thus the 1934 international standards, while being part of the normalized system referred to above, do, however, occupy a special place in the international system by the virtue of the many measurements in which these standards have been involved. Many international secondary standards would in fact only be related to the proposed normalized system through the 1934 Paris and Vienna Standards.

The <u>ad hoc</u> Study Group therefore recommends that the Bureau International des Poids et Mesures should be entrusted with the custody of the 1934 Paris International Radium Standard. It is interesting to note in this connection that the same recommendation was made concerning the 1911 Paris Standard by the Commission Internationale des Etalons de Radium in its meeting in Paris in March 1912.

> With regard to the 1911 Paris International Radium Standard it is recommended, in view of its great historical and technical value, that it could also be placed in the custody of the Bureau International des Poids et Mesures. In this connection it is believed that the appropriate authority to be approached would be the International Council of Scientific Unions, to which the past Joint Commission on Radioactivity, the erstwhile owner of the standard, was responsible through IUPAC and IUPAP. It may be necessary however, to seek legal advice as to the ownership of the 1911 Paris International Radium Standard."

The members of the <u>ad hoc</u> Study Group making these recommendations were G. H. Aston (U.K.), H. Fränz (Germany), M. Frilley (France), B. Karlik (Austria), W. B. Mann (U.S.A.) and J. L. Wolfson (Canada). G. Bourdoun (U.S.S.R.) wrote later to signify the agreement of himself, K. K. Aglintzev and their colleagues of the Radiometric Laboratory of the D. I. Mendeleev Institute of Metrology, provided that the normalized system of standards should include the U.S.S.R. National

Radium Standard that had been acquired by the Soviet Union too recently to be included in the many international comparisons of Hönigschmid standards.

This case history will, I hope, have served to demonstrate the role of the ICRU in the field of international radioactivity standardization, and its close cooperation with the BIPM whose present activities in this field were built very largely on prior ICRU foundation as well as upon its own competence in the field of International Standards. Lastly, through its wide-ranging system of Planning Boards the ICRU will continue to fulfill its mission in the developing field of radiation units and measurements.

## ICRU - Overview

### APPENDIX<sup>1</sup>

# W. B. Mann

## Scope of ICRU Activities

The International Commission on Radiation Units and Measurements (ICRU), since its inception in 1925, has had as its principal objective the development of internationally acceptable recommendations regarding:

- 1. Quantities and units of radiation and radioactivity,
- Procedures suitable for the measurement and application of these quantities in clinical radiology and radiobiology,
- Physical data needed in the application of these procedures, the use of which tends to assure uniformity in reporting.

The Commission also considers and makes recommendations in the field of radiation protection. In this connection, its work is carried out in close cooperation with the International Commission on Radiological Protection (ICRP).

### Policy

The ICRU endeavors to collect and evaluate the latest data and information pertinent to the problems of radiation measurement and dosimetry and to recommend the most acceptable values for current use.

The Commission's recommendations are kept under continual review in order to keep abreast of the rapidly expanding uses of radiation.

The ICRU feels it is the responsibility of national organizations to introduce their own detailed technical procedures for the development and maintenance of standards. However, it urges that all countries adhere as closely as possible to the internationally recommended basic concepts of radiation quantities and units.

Reproduced with permission from the Prefaces of ICRU Report 11 "Radiation Quantities and Units" and Report 12, "Certification of Standardized Radioactive Samples".

The Commission feels its responsibility lies in developing a system of quantities and units having the widest possible range of applicability.

Situations may arise from time to time when an expedient solution of a current problem may seem advisable. Generally speaking, however, the Commission feels that action based on expediency is inadvisable from a long-term view-point; it endeavors to base its decisions on the long-range advantages to be expected.

The ICRU invites and welcomes constructive comments and suggestions regarding its recommendations and reports. These may be transmitted to the Chairman.

## Current Program

In 1962 the Commission laid the basis for the development of the ICRU program over the next several years. At that time it defined three broad areas of concern to the Commission:

- I. The Measurement of Radioactivity
- II. The Measurement of Radiation
- III. Problems of Joint Interest to the ICRU and the International Commission on Radiological Protection (ICRP)

The Commission divided these three areas into nine subareas with which it expected to be primarily concerned during the next decade. The division of work agreed upon is as follows:

- I. Radioactivity
  - A. Fundamental Physical Parameters and Measurement Techniques
  - B. Medical and Biological Applications
- II. Radiation
  - A. Fundamental Physical Parameters
  - B. X Rays, Gamma Rays and Electrons
  - C. Heavy Particles
  - D. Medical and Biological Applications (Therapy)
  - E. Medical and Biological Applications (Diagnosis)
  - F. Neutron Fluence and Kerma

III. Problems of Joint Interest to the ICRU and the ICRP A. Radiation Protection Instrumentation and its Application

The Commission established a separate planning board to guide ICRU activities in each of the subareas. The planning boards after examining the needs of their respective technical areas with some care recommended, and the Commission subsequently approved, the constitution of task groups to initiate the preparation of reports. The substructure which resulted from these actions is given below.

Planning Board I.A.	RadioactivityFundamental Physi-
	cal Parameters and Measurement
	Techniques
Task Group 1.	Measurement of Low-Level Radioacti-
	vity
Task Group 2.	Specification of Accuracy in Cer-
	tificates of Activity of Sources
	for Calibration Purposes
Task Group 3.	Specification of High Activity
	Gamma-Ray Sources (Joint with P.B.
	II.B)
Planning Board I.B.	RadioactivityMedical and Biologi-
	cal Applications
Task Group 1.	In Vivo Measurements of Radioacti-
	vity
Task Group 2.	Scanning
Task Group 3.	Tracer Kinetics
Task Group 4.	Methods of Assessment of Dose in
	Tracer Investigations
Planning Board II.A.	RadiationFundamental Physical
	Parameters
Planning Board II.B.	Radiation-X Rays, Gamma Rays and
	Electrons
Task Group 1.	Radiation Dosimetry; X Rays from 5
	to 150 kV

P

Task Group 2.	Radiation Dosimetry; X and Gamma
	R_ys from 0.6 to 100 MV
Task Group 3.	Electron Beam Dosimetry
Planning Board II.C.	RadiationHeavy Particles
Task Group 1.	Dose As a Function of LET
Task Group 2.	High Energy and Space Radiation
	Dosimetry
Planning Board II.D.	RadiationMedical and Biological
	Applications(Therapy)
Task Group 1.	Measurement of Absorbed Dose at a
	Point in a Standard Phantom (Ab-
	sorbed Dose Determination)
Task Group 2.	Methods of Arriving at the
· · · · · · · · · · · · · · · · · · ·	Absorbed Dose at any Point in a
	Patient (In Vivo Dosimetry)
Task Group 3.	Methods of Compensating for Body
	Shape and Inhomogeneity and of
	Beam Modification for Special Pur-
	poses (Beam Modification)
Task Group 4.	Statement of the Dose Achieved
	(Dosage Specification)
Planning Board II.E.	RadiationMedical and Biological
	Applications (Diagnosis)
Task Group 1.	Photographic Materials and Screens
Task Group 2.	Image Intensifier Radiography
Task Group 3.	TV Systems
Planning Board II.F.	RadiationNeutron Fluence and
	Kerma
Task Group 1.	Neutron Fluence, Energy Fluence,
	Neutron Spectra and Kerma
Planning Board III.A.	Radiation Protection Instrumenta-
	tion and its Application
Task Group 1.	Radiation Protection Instrumenta-
	tion HandbookPart I
Task Group 2.	Neutron Instrumentation and its
	Application to Radiation Protec-
	tion

Because the Commission's basic recommendations on radiation quantities and units relate to the work of all of the planning boards, the Commission decided to establish a separate committee with membership drawn largely from the Commission itself to initiate the revision of ICRU Report 10a, <u>Radiation Quantities and Units</u>. Thus, the Committee on Fundamental Quantities and Units was added to the above substructure.

In 1962 the Commission decided to abandon its past practice of holding a meeting together with all of its subunits every three years. Instead, it was decided that the Commission would receive reports from the subgroups at the time of their completion rather than at fixed deadlines. Meetings of the Commission and of the subgroups are held as needed.

The adoption of the new substructure and mode of operation was intended to alleviate some of the problems associated with the expanded program required in recent years. In the past, the Commission's attempt to administer and review the work of each of the working groups imposed a very considerable burden on the Commission itself. The need to concern itself with each detail, which was inherent in such a scheme of operation, when coupled with the procedure of completing all reports at one time, subjected the Commission members to an intolerable work load if rigorous standards were to be maintained. The new substructure and mode of operation is now beginning to produce results in the form of reports drafted by the task groups and reviewed by the planning boards. Present evidence indicates that the substructure and mode of operation, while not perfect, has to a substantial extent succeeded in alleviating the problems previously experienced. ICRU Reports

In 1962 the ICRU, in recognition of the fact that its triennial reports were becoming too extensive and in some cases too specialized to justify single-volume publication, initiated the publication of a series of reports, each deal-

ing with a limited range of topics. This series was initiated with the publication of six reports:

ICRU	Report	10a,	Radiation Quantities and Units
ICRU	Report	10b,	Physical Aspects of Radiation
ICRU	Report	10c,	Radioactivity
ICRU	Report	10d,	Clinical Dosimetry
ICRU	Report	10e,	Radiobiological Dosimetry
ICRU	Report	10f,	Methods of Evaluating Radiological
			Equipment and Materials

These reports were published, as had been many of the previous reports of the Commission, by the United States Government Printing Office as Handbooks of the National Bureau of Standards.

In 1967 the Commission determined that in the future the recommendations formulated by the ICRU would be published by the Commission itself. This is the second report to be published under this new policy. With the exception of ICRU Report 10a, which was superseded by ICRU Report 11, the other reports of the "10" series have continuing validity and, since none of the reports now in preparation are designed to specifically supersede them, will remain available until the material is essentially obsolete. All future reports of the Commission, however, will be published under the ICRU's own auspices: ICRU Reports, P. O. Box 4869, Washington, D. C. 20008.

ICRU Relationships With Other Organizations

One of the features of ICRU activity during the last few years has been the development of relationships with other organizations interested in the problems of radiation quantities, units, and measurements. In addition to its close relationship with the International Commission on Radiological Protection and its financial relationships with the International Society of Radiology, the World Health Organization, and the International Atomic Energy Agency, the ICRU has also developed relationships of varying intensity with several other organizations. Since 1955, the ICRU has had an official relationship with the World Health Organization (WHO) whereby the ICRU is looked to for primary guidance in matters of radiation units and measurements, and in turn, the WHO assists in the world wide dissemination of the Commission's recommendations. In 1960 the ICRU entered into consultative status with the International Atomic Energy Agency. The Commission has a formal relationship with the United Nations Scientific Committee on the Effects of Atomic Radiation (UNSCEAR), whereby ICRU observers are invited to attend UNSCEAR meetings. The Commission and the International Standards Organization (ISO) informally exchange notifications of meetings and the ICRU is formally designated for liaison with two of the ISO Technical Committees. The ICRU also corresponds and exchanges final reports with the following organizations:

Bureau International des Poids et Mesures

Council for International Organizations of Medical Sciences

Food and Agriculture Organization International Council of Scientific Unions International Electrotechnical Commission International Labor Organization International Union of Pure & Applied Physics United Nations Educational, Scientific and Cultural Organization

Relations with these other international bodies do not affect the basic affiliation of the ICRU with the International Society of Radiology. The Commission has found its relationship with all of these organizations fruitful and of substantial benefit to the ICRU program.

### Operating Funds

Throughout most of its existence, the ICRU has operated essentially on a voluntary basis, with the travel and operating costs being borne by the parent organizations of the participants. (Only token assistance was originally available from the International Society of Radiology.) Recognizing the impracticability of continuing this mode of operation on an indefinite basis, operating funds were sought from various sources in addition to those supplied by the International Society of Radiology.

Prior to 1959, the principal financial assistance to the ICRU had been provided by the Rockefeller Foundation which supplied some \$11,000 to make possible various meetings. In 1959 the International Society of Radiology increased its contribution to the Commission providing \$3,000 for the period 1959-1962. For the period 1962-1965 this was again increased, the Society providing \$5,000. In 1960 the Rockefeller Foundation supplied an additional sum of some \$4,000 making possible a meeting of the Quantity and Units Committee in 1960.

In 1960 and 1961 the World Health Organization made available the sum of \$3,000 each year. This was increased to \$4,000 in 1962 and this amount has been made available annually since then. It is expected that this sum will be allocated annually, at least for the next several years.

In connection with the Commission's Joint Studies with the ICRP, the United Nations allocated the sum of \$10,000 for the joint use of the two Commissions.

The most substantial contribution to the work of the ICRU has come from the Ford Foundation. In December 1960, the Ford Foundation made available to the Commission the sum of \$37,000 per year for a period of five years. This grant was to provide for such items as travel expenses to meetings, for secretarial services and other operating expenses. In 1965 the Foundation agreed to a time extension of this grant making available for the period 1966-1970 the unused portion of the original grant. To a large extent, it is because of this grant that the Commission has been able to move forward actively with its program.

In 1963 International Atomic Energy Agency allocated the sum of \$6,000 per year for use by the ICRU. This was increased to \$9,000 in 1967. It is expected that this sum

### ICRU - Overview

will be allocated annually at least for the next several years.

From 1934 through 1964 valuable indirect contributions were made by the U. S. National Bureau of Standards where the Secretariat resided. The Bureau provided substantial secretarial services, publication services and travel costs in the amount of several thousands of dollars.

The Commission wishes to express its deep appreciation to all of these and other organizations that have contributed so importantly to its work.

### Composition of the ICRU

It is of interest to note that the membership of the Commission and its subgroups totals 140 persons drawn from 16 countries. This gives some indication of the extent to which the ICRU has achieved international breadth of membership within its basic selection requirement of high technical competence of individual participants.

The membership of the Commission during the preparation of this report was as follows:

Lauriston S. Taylor, Chairman

M. Tubiana, Vice Chairman

- H. O. Wyckoff, Secretary
- A. Allisy
- J. W. Boag (1965-1966)
- R. H. Chamberlain
- F. P. Cowan
- F. Ellis (1965)
- J. F. Fowler
- H. Franz (1965)
- F. Gauwerky
- J. R. Greening
- H. E. Johns (1965-1966)
- K. Liden
- R. H. Morgan
- V. A. Petrov (1965)
- H. H. Rossi

# ACTIVITIES OF THE SUBCOMMITTEE ON USE OF RADIOACTIVITY STANDARDS, NAS-NRC Bernd Kahn

The activities of the subcommittee and some of its experiences with radioactivity standards are discussed. The subcommittee consists of individuals active in preparing or using such standards. It was formed to improve the availability of radioactivity standards by discussing the need for specific radionuclides, and the degree of accuracy and extent of information required for each standard. These needs are communicated to suppliers, round-robins have been performed to test and improve accuracy, and a standard certificate was distributed to commercial suppliers. To guide the appropriate use of radioactivity standards, the subcommittee has prepared short reviews of chemical and counting problems, and encouraged the preparation of simple "best" decay schemes for frequently used standards. Suggestions for additional activities are welcomed.

Twenty years ago, most radioactivity standards were used by nuclear physicists and radiochemists in elucidating nuclear decay characteristics; today, these standards are applied in fields as diverse as nuclear reactor technology, nuclear medicine, and public health. The National Bureau of Standards continues to supply several radioactivity standards, and also calibrates certain radioactivity sources submitted to it.[1] Most radioactivity standards in the United States, however, are supplied by commercial producers. As an indication of the magnitude of the supply, eight commercial suppliers offered standards in 1967,[2] and 70 radionuclides were listed as available standards in a review published that year.[3] The availability of numerous standards, and their use by many with limited pertinent experience, led the Committee on Nuclear Science of the NAS-NRC in 1962 to appoint the Subcommittee on 49

#### NAS-NRC

Use of Radioactivity Standards as successor to the Subcommittee on Measurements and Standards of Radioactivity. The change in names indicates the shift in emphasis.

Improvement in standardization techniques during the past twenty years is evident from reports published in proceedings of three topical symposia [4,5,6] and in NBS Handbook 86 [7]. Briefly, absolute standardization through  $4\pi$  beta and X-ray counting and beta-gamma and gamma-gamma coincidence counting has been extended to radionuclides that decay only by K-capture, through emission of low-erergy beta spectra, and via complex beta- and gamma-branching. Absolute standardization, moreover, is now applied with greater confidence in reproducibility. Relative ratings -- frequently by scintillation spectrometry or high-pressure ionization chamber -- are also believed to be more reliable because of accumulated experience, availability of standards over a wide range of energies and stable detection devices. The 2-sigma error for many radionuclides is now considered to be 0.1 to 0.5 percent by meticulous and highly skilled absolute calibration, and near 2 percent by competent routine measurements.

The extent of problems concerning radioactivity standards is difficult to evaluate, since such information is mostly by hearsay. On the basis of two reports [8,9] and individual complaints over a period of years, the major problem is a lack of interest by a few commercial suppliers in preparing, rating, and certifying standards with the meticulousness required for use of that term. Other common problems are typified by the following situation: two standards obtained from different suppliers at an interval of several months to calibrate a detector for radiopharmaceuticals differ by 15 percent, although each is rated  $\pm$  3 percent. Was the supplier at fault through bad measurement, wrong error term, or inclusion of impurities? Or was the user at fault through inappropriate handling of the standard or poor instrument maintenance?

### Kahn

The Subcommittee was formed to survey existing problems and to formulate remedies. By appointing to membership professionals who prepare commercial standards, representatives of national standardization laboratories, and frequent users, the Subcommittee has obtained information, evaluated improvements, and developed aids to users from both the supplier's and the user's point of view. The program evolved from this cooperation is summarized in Table 1.

As its first activity, the Subcommittee surveyed users' needs to obtain information for planning its program. Questionnaires were distributed to purchasers of radionuclide solutions from AEC contractors, users of radioactivity standards known to Subcommittee members, and state health departments. The response was disappointing in that no concensus existed concerning needs, and no needs unknown to the Subcommittee were uncovered. It was apparent that a major difficulty in procuring satisfactory radioactivity standards was the individual requirements set by many of the users. The other five activities listed in Table 1 were planned in response to indicated problems.

A standard certificate [10] was prepared by three members of the Subcommittee to remind commercial suppliers of the type and extent of information required by various users of radioactivity standards. The certificate makes available detailed information concerning the preparation,

TABLE I

Subcomm	ittee	Activ	ities

- 1. Survey of Users' needs
- 2. Preparation of recommended certificate of radioactivity standards
- 3. Proposal to evaluate accuracy of radioactivity standards
- 4. Recommendation to compile "best" decay schemes
- 5. Preparation of guides for using radioactivity standards
- Sponsoring interlaboratory comparisons of radionuclide concentration measurements

### NAS-NRC

composition, and standardization of solutions, and of the magnitude, composition, and change with time of the error in rating. The primary purposes of the certificate are transmission of information and reduction of misunderstandings between supplier and user. It is also intended to lead to a clearer distinction than now exists between a carefully prepared radioactivity standard and a routinely calibrated solution, since the latter would not be accompanied by a detailed certificate. The standard certificate has been distributed to the known commercial suppliers of radioactivity standards in the U. S., and five suppliers have indicated their intention to follow its pattern. Copies of the certificate can be obtained from the Subcommittee.

At the request of the Subcommittee, a program for evaluating the accuracy of commercially available solution of radioactivity standards was prepared.[11] An independent laboratory would purchase replicate standards and carefully determine their radionuclide concentration by absolute measurement. Comparison of measured values and standard deviations among replicates with the supplier's rated values and error terms would indicate the degree of reliability of the standards. The proposal estimates the number of replicates needed for stated degrees of confidence, and the cost of the evaluation. If the project is to receive financial support, it will undoubtedly be necessary to demonstrate that the cost of the project is less than the expense incurred in independent calibration by users who distrust purchased standards.

A compilation of best decay characteristics was recommended by the Subcommittee to provide consistent values for calibrating and using radioactivity standards. The Nuclear Data Group under the direction of K. Way accepted this suggestion in 1967, and is preparing a publication that should be available within a year.[12] It will have the same general format as the earlier publication by Slack and Way.[13] It is to contain what have been evaluated by its authors to be the best half lives, energies, intensities, and conversion

coefficients for approximately 100 radionuclides in relatively common use. Dosimetry information will also be included.

Summaries of the more important chemical and counting problems in utilizing radioactivity standards were prepared by Subcommittee members as aids to the memories of users.[14] Potential losses during dilution and evaporation, interference from isotopic impurities, effects of genetic relation between radionuclides, the influence on counting rates of low-energy beta groups, internal conversion, and branching, etc., are briefly mentioned. Each of 40 elements is discussed on a single page, and references to more detailed information are provided. Copies of the users' guides have been distributed to commercial suppliers and are available from the author.

Finally, subcommittee members have participated in distributing radionuclide solutions to several laboratories (including commercial producers) and comparing measured concentration values. Results of the first three sets of measurements sponsored by the Subcommittee are summarized in Table 2. Note the close agreement of absolute values, the agreement between the means of absolute and relative values, and the much wider range for relative values than would be predicted for the usually accepted 2-sigma value of approximately 2 percent. The range of values in this type of comparison indicates the state of the art. Agreement among several absolute values confirms the individual values, and enables participants who determine activity relatively to improve their counter calibration. Subcommittee members will continue to participate in comparisons for less common radionuclides as they become of interest as standards, or when serious inconsistencies arise in available standards.

Future activities depend on the needs of users, and users are urged to make these known to the Subcommittee. Among categories of needs that should be considered are stimulation of new developments in standardization techniques, availability of more radionuclides as standards, preparation of more accurate standards, and dissemination of additional information to users of radioactivity standards.

### Kahn

### TABLE 2

Interlaboratory	Comparisons

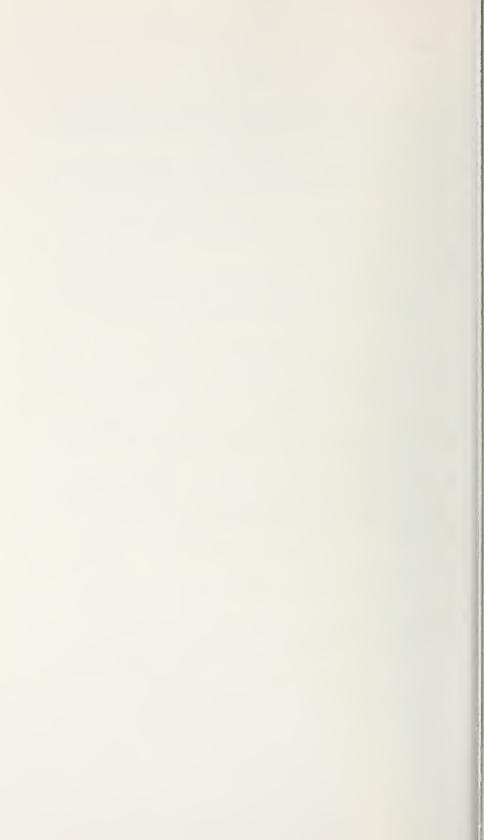
	No. of Participants	57 <sub>Co</sub>	99 <sub>Mo</sub>	99m <sub>Tc</sub>
Absolute values $(4\pi\beta-\gamma)$	2			
range/mean	2	0.9%	0.6%	0.2%
Relative values $(\gamma)$ ,				
range/mean	10	21. %	23. %	21. %
Ratio of mean values,				
relative/absolute		1.03	1.00	0.98

Note: Standard deviation of mean is approximately 0.61 of range for 2 values and 0.31 of range for 10 values.

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- [7] "Radioactivity Recommendations of the International Commission on Radiological Units and Measurements (1962) (ICRU) Report 10c", NBS Handbook 86 (U. S. Government Printing Office, Washington, D. C.).
- [8] H. E. Kolde and G. J. Karches, "Some Limitations of Available Radioactivity Standards", Health Phys. <u>10</u>, 635 (1964).
- [9] H. E. Kolde and P. A. Cliggett, "Radioactivity Source Standards for Public Health Service Needs", 156th ACS National Meeting (1968).
- [10] S. B. Garfinkel, A. P. Baerg, and P. F. Zigman, "Certificates of Radioactivity Standards", (National Academy of Sciences-National Research Council, Washington, D. C., 1966).
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- [13] L. Slack and K. Way, "Radiations from Radioactive Atoms in Frequent Use" (U. S. Atomic Energy Commission, Washington, D. C., 1959).
- [14] B. Kahn, G. R. Choppin, and J. G. V. Taylor, "Users' Guides for Radioactivity Standards" (National Academy of Sciences-National Research Council, Washington, D. C. 1967).

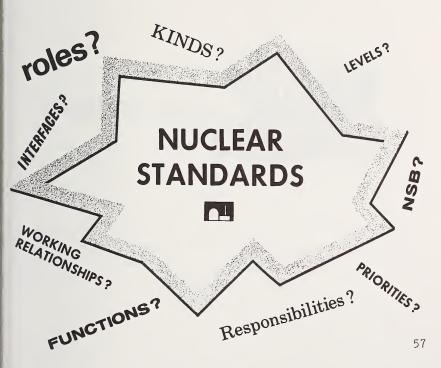


# STANDARDS PROGRAM OF THE AMERICAN NUCLEAR SOCIETY

#### R. G. Chalker

The American Nuclear Society is a nuclear oriented interdisciplinary society organized into 10 professional divisions. Many nuclear chemistry and technology division members of the audience are members of the American Nuclear Society along with fellow professionals in other technical disciplines. Thus, because of the natural make-up of the American Nuclear Society it has become a focal point for nuclear standards.

I will discuss some of the problems experienced in ANS over the past 12 years in generating nuclear standards with the intent that they may be of some help to others engaged in similar activity.



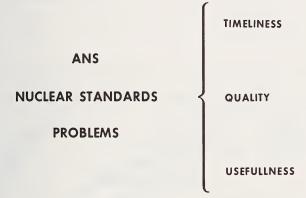
Interest in nuclear standardization is currently very intent in all segments of the industry. One of the principal reasons for this high interest is the potential threat that government regulations may preempt industry standards. This and other elements caused the Executives and Board of Director of the American Nuclear Society to reevaluate standards

# **Government Preemption?**

activities within the Society.



Careful analysis of industry and governmental criticism of the nuclear standards program revealed that there were three principal problems with the nuclear standards program. These problems indicated in Chart 3 will be discussed in detail



As a result of the analysis, the ANS Board of Directors approved a new policy rededicating itself to a more vigorous program for the development of nuclear standards. The policy contains four principal elements as noted. Element I declares the desirability of standards being generated by industry specialists who are actively working in the field. These men are best qualified to indicate practical procedures, guides and practices ready for meaningful standard ization and are best qualified to write such standards.

Element II recognizes the delays inherent with using the consensus principle for adopting standards but believes that within these constraints streamlining is possible.

Element III reflects personal competence and interest not only by the Standards Committee but also by members at large, the Society President, and Board of Directors in nuclear standards and offers this talent to others to assist in relate endeavors.

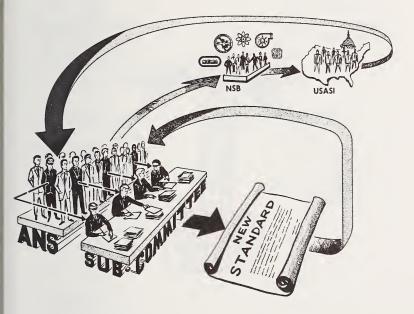
Element IV recognizes the desirability of maintaining an independent society activity in standards generation along wit an awareness that cooperation with other societies and the government is necessary.

# ANS STANDARDS

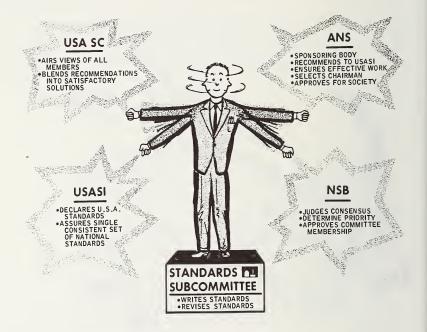
- 1. WE REAFFIRM THE DESIRABILITY OF THE CONSENSUS APPROACH TO THE DEVELOPMENT OF NUCLEAR STANDARDS, BUT ALSO RECOGNIZE THE DIFFICULTY IN THIS METHOD OF MEETING SHORT-TERM REQUIREMENTS ON NEW ISSUES.
- 2. WE PROPOSE TO CONTINUE THE CONSENSUS APPROACH IN COOPERATION WITH USASI AND WITH OTHER PROFESSIONAL SOCIETIES AND WILL VIGOROUSLY EXPLORE MEANS OF STREAMLINING PROCEDURES.
- 3. WE BELIEVE THAT THE TALENTS REPRESENTED IN THE ANS AND THE ORGANIZATIONAL STRUCTURE IN OUR STANDARDS COMMITTEE AND ITS SUBCOMMITTEES CAN BE EMPLOYED USEFULLY TO ASSIST THE AEC AND OTHER GOVERNMENT AGENCIES IN MEETING URGENT PROBLEMS.
- 4. WE BELIEVE THAT THE ANS HAS A RESPONSIBILITY TO MAINTAIN AN INDEPENDENT BUT COOPERATIVE ROLE IN THE GENERATION AND ADOPTION OF NUCLEAR STANDARDS.

#### Chalker

The reason for lengthy in-process time is inherent with the theory of involvement of all principal interests in agreeing to a standard so that it will have maximum chance for voluntary acceptance when issued. The process for adopting a J.S.A. Standard involves review and judgment of various organizations each representing a larger segment of the industry until finally within USASI the entire country has a representative voice.



Basic to the streamlining of the consensus gathering procedure has been the problem of intermixing the standards writing and adoption process. The ANS Standards Committee procedures have now been revised to separate these two processes. This allows the writers to concentrate on producin useful, adequate standards and leaves the complex interfaces and judgments of the adopting process to others.



Chalker

One method of streamlining the consensus gathering procedures without destroying the basic desirability of the principle was to eliminate some reviews. Fundamental to the subsequent review deletions was the recent selection of the American Nuclear Society to become sponsor of the N-16 and N-18 U.S.A. Standards Committee. The importance of this development is that the scopes of these two committees embrace the majority of standardization work within the Society, thus making it possible to reorganize the committee as will be shown.

# N-16-NUCLEAR CRITICALITY SAFETY

# SCOPE

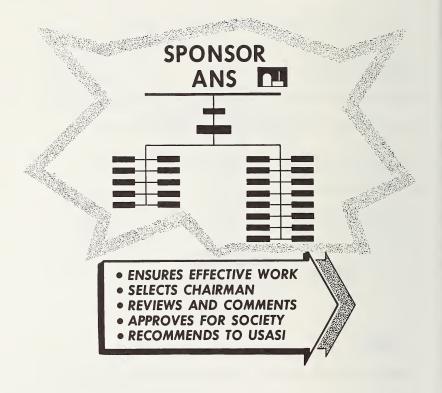
STANDARDS FOR DETERMINING THE POTENTIAL FOR NUCLEAR CRITICALITY OF FISSILE MATERIALS OUTSIDE REACTORS, FOR THE PREVENTION OF ACCIDENTAL CRITICALITY, AND FOR COPING WITH ACCIDENTS SHOULD THEY OCCUR.

# N-18-NUCLEAR DESIGN CRITERIA

# SCOPE

NUCLEAR ASPECTS OF DESIGN CRITERIA FOR NUCLEAR FACILITIES

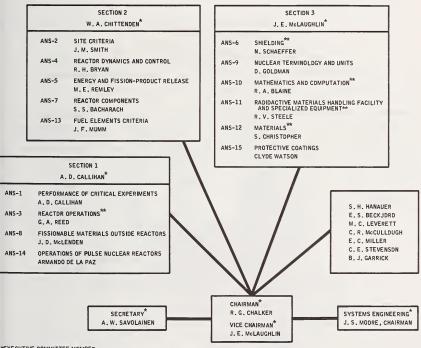
The new procedures now in effect take advantage of this organization structure so that any standard written by subcommittees of the American Nuclear Society intended to be processed eventually as a U.S.A. Standard will be processed directly from the American Nuclear Society into the appropriat sectional USASI standards committee, thus eliminating the internal American Nuclear Society reviews and reducing the standards adoption process to a minimum.



ANS

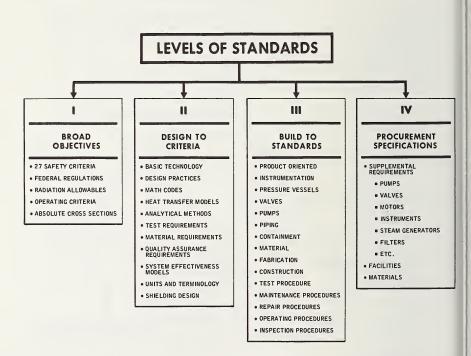
#### Chalker

The Standards Committee was reorganized into three sections. Section 1 is composed of existing American Nuclear Society subcommittees whose individual scopes of nuclear standardization fall within the general scope of N-16 -- Nuclear Criticality Safety. Similarly, Section 2 has those subcommittees whose activities fall within the scope of N-18 -- Nuclear Design Criteria. Section 3 is composed of subcommittees with scopes falling outside these two U.S.A. Standards Committee scopes.



# ANS STANDARDS COMMITTEE

\*EXECUTIVE COMMITTEE MEMBER \*\*SUBCOMMITTEE SPONSORED BY AN ANS DIVISION An important element in speeding the standardization process is to establish priorities on those standards urgently needed and assure that complementing standards are processed approximately simultaneously. Once priorities are established and interfacing standardization organizations are properly identified, work can proceed systematically. Standards have been categorized into four levels. The writing of standards will progress more rapidly if priorities are given to level I so they will set requirements for Group II. Subsequently, Group II sets requirements for Group III, etc.



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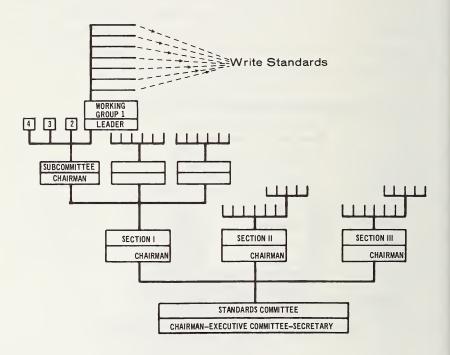
Improved quality can be achieved in nuclear standards by assuring that those professionals capable in the field are being selected to write the standards. Within the American Nuclear Society's professional divisions, five are sponsors of standards writing subcommittees within the scope of their expertise. Thus, the organizational structure of the Society naturally provides a reservoir of qualified professionals from whom a select few may be chosen for a writing assignment. The product of the writing subcommittee then represents the best information continuously being developed through normal professional activities. Subsequent to the concurrence of the subcommittee, the standard is reviewed by a 3-man ad hoc committee chosen from professional experts at large.



This organization chart of the ANS Standards Committee graphically shows the importance of the subcommittee writers in producing quality standards. The writers are on top and all others are part of the adoption process.

ANS

Useful standards then are dependent on good standards process via the consensus process in the most expeditious manner.



Indicative of the Society's policy to cooperate with government agencies is a proposal offered by the President of the American Nuclar Society to the Chairman of the U.S. Atomic Energy Commission.

# PROPOSED ANS INTERFACE WITH AEC

- 1. EXCHANGE OF PLANNING AND TECHNICAL INFORMATION IN THE FORMATIVE STAGES OF NUCLEAR STANDARDS DRAFTING AND CONTINUING DURING THE ENTIRE STANDARDIZATION PROCESS
- 2. AD HOC TECHNICAL REVIEW SERVICE FOR LICENSING CRITERIA
- 3. SENIOR REVIEW PANEL TO CONSIDER SOME OF THE BROADER OBJECTIVES AND TRENDS OF LICENSING AND REGULATORY PROGRAMS

The American Nuclear Society has more than 150 members working on 29 nuclear standards drafts in addition to 10 standards already written and approved. The following list shows some of the standards which may be of interest to the American Chemical Society members.

	STANOARO	ANS APPROVEO STANOARO	USASI APPROVEO STANDARO	STANOARO SUBMITTEO TO USASI FOR APPROVAL	SURVEY BEING CONDUCTEO FOR NEEO	ORAFT	NUCLEAR ENGINEERING BULLETIN
1.	CODE OF GODO PRACTICES FOR THE PERFORMANCE OF CRITICAL EXPERIMENTS	x					
2.	STANDARO VALUES FOR ENERGY RELEASE FOLLOWING SHUTOOWN OF URANIUM- FUELED THERMAL REACTORS					x	
3.	SUMMARY OF FISSION YIELOS FOR 235U, ANO 239Pu AT THERMAL FISSION SPECTRUM, ANO 14-MeV NEUTRON ENERGIES					x	
4.	STANDARD HALF-LIVES FOR FISSION PRODUCTS					x	
5.	STANOARD FOR FISSION PRODUCT YIELOS					x	
6.	POST-SHUTOOWN HEAT GENERATION AND TEMPERATURE INCREASE RATES IN U02 FUELED CORES					×	
7.	STANDARD TEST PROGRAM FOR BIOLOGICAL SHIELOING IN NUCLEAR REACTOR PLANTS						×
В.	RECOMMENCED CATA FOR SHIELDING CALCULATIONS				×	1	
9.	SPECIAL MATERIALS FOR USE IN REACTOR SHIELOS				x		
10.	STANOARO FOR PLATE-TYPE URANIUN -ALUMINUM FUEL ELEMENTS	x		x			
11.	STANOARO GUIOE FOR SPECIFYING CLEANLINESS IN NUCLEAR REACTOR SYSTEM COMPONENTS	×					
12.	STANOARO FOR QUALITY CONTROL AND INSPECTION FOR PLATE-TYPE URANIUM- ALUMINUM FUEL ELEMENTS		x				
13.	STANOARO FOR NUCLEAR FUEL ELEMENTS FOR POWER REACTORS					×	
14.	A SAFETY STANDARD FOR OPERATIONS WITH FISSIONABLE MATERIALS OUTSIDE REACTORS		x	1			
15.	STANDARO FOR USE OF BOROSILICATE-GLASS RASCHIG RINGS AS A FIXEO NEUTRON ABSORBER IN SOLUTIONS OF FISSILE MATERIAL						x
16.	STANOARD ON THE USE OF BORON STEEL AS A FIXED NEUTRON ABSORBER					×	
17.	STANOARO ON THE SOLUBLE NEUTRON ABSORBERS IN PROCESS SOLUTIONS					x	
1B.	SHIPPING CONTAINERS FOR URANIUM HEXAFLOURIDE				x		
19.	STANDARD NUCLEAR REACTOR CLASSIFICATION			×			x
20.	STANGARG TERMS AND UNITS FOR IONIZING RADIATION					x	
21.	RADIOACTIVE MATERIAL FLOW SCHEMES	1					
	OVERALL TOTAL STANOAROS	5	2	3	3	24	6

# SELECTED ANS STANDARDS

# THE POSITION OF ASTM IN NUCLEAR TECHNOLOGY J. W. Caum

The American Society for Testing Materials actually had its beginning in 1898 when an American section of the International Association for Testing Materials was organized. The IATM was dedicated to "the development of standard methods of testing for the determination of the properties of the materials of construction and of other materials, and also the perfection of apparatus for that purpose." All European nations except Turkey participated, with Germany and Russia the big wheels. In 1898 the total membership was about 1500 with about 70 U.S.A. members. A noteworthy feature regarding membership (one that has carried through to the present in ASTM) was that it could be assumed by a corporation or society as well as a person. The first list of American members included the Franklin Institute, the American Society of Mechanical Engineers, the American Foundrymen's Association, local engineering clubs, several steel companies, engineering journals, and firms engaged in inspection and testing. In Europe such governmental bureaus were members as the public works of several cities, provinces, and states, police bureaus, and war departments. ASTM now includes many federal, state, and local agencies on its membership rolls as well as governmental agencies of many other nations around the world. The IATM early stated that such an arrangement makes it possible for both manufacturers and consumers to make their wishes more directly known and thus differences in regard to methods of inspection and testing can be more quickly harmonized than under the usual plan of strict individual membership.

After several years of operation as an American Section, it was decided to incorporate as an independent self directing but closely related Society. Thus in 1902 ASTM was formally incorporated for "the promotion of the knowledge of

#### ASTM-Overview

the materials of engineering and the standardization of specifications and methods of test." This step was taken for two reasons. First our independent democratic nature created a fundamental difference of opinion with the Europeans. We disagreed with the premise that the IATM Council could itself appoint the American member of the Council. We felt that we should appoint our own representative. Second the American Section early recognized the primary importance of establishing the specifications which the materials tested must endure (purchase specifications) and this was shrugged off by the IATM. Although incorporated as ASTM we continued to be recognized as the American section of IATM until its dissolution in 1925. We have maintained our international aspects with 2000 of our total 15,000 members being located outside the U.S.A. and with many of our technical committees furnishing guidance to USASI in international standards activities. In response to requests from our European members we have sponsored ASTM meetings in Europe.

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Early in our history our standardization program was aimed primarily at railroad needs (the prime consuming industry). This continued until the late 1940's when the requirements of the power industry replaced railroads as the primary need. Then nuclear power came into the picture in the 1950's. Let me hasten to add that many other industries' needs for standards have been fulfilled over the years. I am merely pointing out the largest portion of ASTM activities in mentioning railroads and electric power.

During the term of office of C. H. Fellows, Detroit Edison Company, as ASTM President, both ASTM and ASA (the predecessor of USASI) held conferences late in 1955 on nuclear energy. At that time it was established that ASTM should be the central focus for material standards. In January 1956 the ASTM formed a Special Administrative Committee on Nuclear Standards to promote standardization work in the nuclear energy area within the ASTM technical committees and to coor-

#### Caum

dinate nuclear energy matters within ASTM and with other organizations. The members of this group were specialists appointed by the ASTM Board of Directors, and its function was administrative in character rather than the actual writing of standards. During the succeeding years this group did interest numerous existing ASTM technical committees in expanding their activities into the nuclear field and also brought to light numerous existing ASTM test methods and specifications that were adaptable to the nuclear energy field. Close relations were maintained with the American Nuclear Society, ASA, and ASME, particularly in code work for pressure vessels and pressure piping for nuclear energy became involved in ASTM technical committee work.

By the early part of 1965 a decision was reached that this Special Committee of the Board of Directors had served its purpose. Therefore in May, 1965, the ASTM Board of Directors dissolved the Special Committee and authorized a Coordinating Committee on Materials Specifications for Nuclear Service. This coordinating committee comprises the chairmen or key representatives of all ASTM technical committees or subcommittees having specific interests in the nuclear field with a specially selected number of individuals to act in an advisory capacity. To match the industrial maturity attained in the nuclear field the primary functions were to (1) coordinate specification activities in ASTM; (2) act as ASTM clearing house for information on nuclear materials technology and specifications to industry, and (3) serve as ASTM liaison to Section III on Nuclear Vessels of the ASME Boiler and Pressure Vessel Committee. Currently there are 18 ASTM technical committees represented on this coordinating committee, and you will hear about the work of some of these committees in detail in other presentations.

In 1966 the USASI Nuclear Standards Board undertook the reorganization of the N series of USA Standards Committees. During the reorganization ASTM was asked and agreed to spon-

#### ASTM-Overview

sor USA Standards Committee N 11 on Basic Materials and Materials Testing Involved in Nuclear Applications. The scope of this committee is "Standards for the specification of chemical composition and physical and mechanical properties of materials used in or resulting from nuclear applications, and methods of testing and analysis of these materials but not to include the specification and testing of components made from materials." All of the members of the previously mentioned ASTM coordinating committee are either voting or consulting members of N ll. Fifteen other organizations are members of N ll as well as 6 individual persons. It is generally believed that N ll could serve the assigned functions of the ASTM Coordinating Committee as well as many others, Therefore the ASTM Coordinating Committee may be dissolved in the next few years. As sponsor we regard N ll as not primarily a standards writing body but one which would be an excellent judge as to the suitability of standards developed by existing standards-writing organizations for adoption as USA standards. Of course if no existing standards writing organization is capable or willing to undertake a specific task, N 11 could develop a U.S.A. standard itself. This procedure is in accordance with the Constitution and Bylaws of the USA Standards Institute.

While we are on the subject of USA Standards Boards and USA standards in general, I should point out that ASTM is the major contributor to the list of USA standards. It is the official policy of ASTM to refer its standards to USASI for consideration as USA standards under the Existing Standards Procedure --- currently 1400 of 3300 ASTM standards are USA standards or are under consideration as such. I do not have an accurate count at present of the total number of USA standards, but ASTM contributes at least one-third the total number. Becoming more specific, many ASTM standards are essential references in Section III on Nuclear Vessels of the ASME Boiler and Pressure Vessel Code as well as the relatively new AEC-RDT Standards issued by the Division of Reactor Development and Technology from Oak Ridge.

#### Caum

The Society since 1898 has continued to expand and consolidate its ideals, philosophies and procedures as early enumerated by our founders. Whenever the demand for standardization in a new area arises, and the Society can identify sufficient support among producers, consumers and general interest groups, a new project either in existing committees or new committees is created to write the needed standards. In the process of forming a new project, and indeed in the process of sustaining the vigor of existing committees, ASTM uses every facility at its command to seek out and bring into the work every individual and every organization that has an interest. Often identified as the largest private standardization effort in the world, our procedures require a consensus of all concerned and have been thoroughly tested by time, usage, and the courts. To ensure that its standards are unbiased, ASTM requires that the membership of any specifications writing committee be balanced between the producers on one hand and the consumers and general interest groups on the other. In order to achieve the broad consensus required for the adoption of an ASTM standard, all negative votes are thoroughly explored and, if possible, resolved. It is on these provisions of fair play in ASTM committees that the world-renowned integrity of ASTM standards is built.

Drafting a standard in a committee of many interests is not an easy task, as many of you know. The time required for the development of a standard is directly related to the time contributed by the committee members. And, as all of us know, the time made available for committee work is directly proportional to the relative importance of the work to a man's company. There is also the matter of priority of work within one's own company and the fact that each day is composed of a fixed number of hours. The dedicated engineers and scientists who have taken upon themselves the responsibility for the continuing growth, validity and usefulness of ASTM standards have my utmost respect, particularly those who handle the secretarial chores.

#### ASTM-Overview

For quite a few years the ASTM Board of Directors has realized that the chores facing the secretary of an ASTM technical committee are becoming too much for any one company to absorb in relation to amount of time and cost. Therefore plans are now underway for ASTM Headquarters to assume the jobs of maintaining committee mailing lists, preparing, duplicating and mailing minutes and ballots; counting and reporting results of letter ballots, and in general maintaining committee records and files. These functions would be absorbed only for the main technical committee. The secretarial chores for subcommittees would remain a contributed effort.

For some 70 years ASTM technical committees and joint committees with other associations have proliferated on an average of about two per year on a completely horizontal basis. With the advancement and overlapping of technologies such a growth has created quite a coordination problem for the Board of Directors to whom all technical committees are subservient in their scope of activities. The Society is seriously considering a total restructuring of its committees into technological areas of mutual interest. Each technological area might have a technical board of its own to furnish the needed executive direction for activities in this area. One area under active consideration is that of plastics and rubber with all the pertinent composite materials, old and new, that have come into the industrial picture.

In conclusion I want to emphasize that ASTM has served any industry that feels the needs for test methods and specifications for materials and is willing to contribute the time of appropriate industry personnel for the task. The pace of the development of standards and the acceptability of the standards depends entirely on the time that the companies make available for individuals in this effort and the quality of the individuals. ASTM furnishes the structure and the guidance for the development of acceptable standards but the nuclear industry itself must accept the responsibility for the extent of the effort.

# ASTM C-21, SUBCOMMITTEE V, NUCLEAR APPLICATIONS Harlan J. Anderson INTRODUCTION

Subcommittee V was organized in 1962 with a scope as shown in Table I, to study ceramics for nuclear applications. The parent committee is ASTM C-21, "Ceramic Whitewares and Related Products." Since 1962, the group has been active especially in the study of ceramics used in nuclear fuel applications. When the American Standards Association (ASA) was reorganized, Subcommittee V was expanded to include nuclear fuel specifications work, and many of the former ASA N5.1 members are now included in the present membership. It now has 24 active members, excluding the BeO Task Group membership.

# TABLE I

## ASTM SUBCOMMITTEE V (C-21)

- TITLE: Nuclear Applications.
- <u>SCOPE</u>: The study of ceramic materials for nuclear applications; to develop standards to apply to all areas of nuclear ceramics; to promote and encourage mutual cooperation with government agencies, industrial firms, and testing laboratories.

## BACKGROUND

Initially, the Subcommittee studied and reviewed private and governmental specifications of ceramic materials that were being used or proposed for use as nuclear fuels in thermal reactors. The study indicated that the use of uranium oxide (urania) as a fuel was becoming well developed and established. It was apparent that minimum specifications for uranium and uranium oxides should be developed.

## ASTM-Nuclear Fuels

#### PRESENT STANDARDS

Four basic specifications or standard procedures were developed and proposed for standards. Table II shows these standards for uranium, uranium dioxide(s) and related tests. These are:

- Specification for Nuclear Grade Uranium Metal Melt Stock
- (2) Specification for Nuclear Grade Uranium Dioxide, Sinterable
- (3) Specification for Nuclear Grade Uranium Dioxide, Compactible
- (4) Referee Methods for the Chemical Analysis of Nuclear Fuels (Uranium).

These were approved and issued in September 1965. These standards are now also approved by the United States of America Standards Institute (USASI).

#### CURRENT STANDARD ACTIVITIES

Recently, the Subcommittee recognized that while ceramic fuel materials have been studied primarily for thermal reactors, recent expanding nuclear technology as related especially to fast reactor concepts are focusing attention on additional ceramic fuel materials, such as plutonium and plutonium oxides. In addition, interest in ceramic materials such as carbides and nitrides for use as fast reactor nuclear fuels is rapidly expanding. To meet these needs of industry and government, the Subcommittee has been working on several proposed standards. These are shown in Table III. In particular, activities for standards of Nuclear Grade Plutonium Metal; Nuclear Grade Plutonium Dioxide, Sinterable; Referee Methods for the Chemical Analysis of Plutonium-Bearing Fuels; and two referee tests for density and organic impurity in nuclear fuel are in active work status. The Nuclear BeO is also in active status and is undergoing ballot by the Task Group BeO, which is sponsored by Subcommittee V.

TABLE II

ASTM SUBCOMMITTEE V (C-21), "NUCLEAR APPLICATIONS"

Remarks	Issued	Issued	Issued	Issued
Date of Standard	9-65	9-65	9-65	9-65
Status	Approved	Approved	Approved	Approved
Standard Number	N5.4-1965 Approved	N5.5-1965 Approved	N5.6-1965 Approved	N5.7-1965 Approved
Standards Activities	• Specification for Nuclear Grade Uranium Metal Melt Stock	<ul> <li>Specification for Nuclear Grade Uranium Dioxide, Sinterable</li> </ul>	<ul> <li>Specification for Nuclear Grade Uranium Dioxide, Compactible</li> </ul>	<sup>o</sup> Referee Methods for the Chemical Analysis of Nuclear Fuels

TABLE III

ASTM SUBCOMMITTEE V (C-21), "NUCLEAR APPLICATIONS"

Remarks	Fourth draft	Sixth draft	Second draft	Currently under preparation	Second draft	Ballot
Status	Active	Active	Active	Active	Active	Active
Standards Activities	° Nuclear Grade Plutonium Metal	Nuclear Grade Plutonium Dioxide, Sin- terable	<ul> <li>Analytical Method for Determining Organic Material in Nuclear Fuel</li> </ul>	<sup>o</sup> Referee Methods for the Chemical Analysis of Plutonium-Bearing Fuels	<sup>o</sup> Referee Methods for Determining the Density of Solid Fuel Materials	° Nuclear BeO

#### Anderson

Due to increased interest by industry and government, these standards are being reviewed extensively by the Subcommittee members as shown in Table IV. It was evident from Table III that some standards have been issued as drafts and reviewed as many as four or six times within the Subcommittee. This action stems from new technology and new interests about these standards. One particular problem is to overcome the issue and approval of an "ideal" standard. By this I mean a standard that covers all interests of industry and government as well as the criteria of a perfect specification. For example, often not everything is completely known about a ceramic material! However, by using a minimum specification approach and by holding at least two meetings a year preceded by committee letters and drafts, progress is being made. Additionally, specific industrial specifications can be made from these minimum standards.

Hopefully, several of these specifications should be completed and recommended for ballot late this year. Later on in this text, a discussion is given about a proposed activity that will speed up this process of developing standards to meet industrial and governmental needs.

#### LIAISON ACTIVITIES

Additional activities of the Chairman included liaison with the ASTM C-21 Task Group on Beryllia and with other related standardization committees. In particular, liaison activities were conducted with the former ASA N5.1 Committee, and with the ASTM Coordinating Committee on Materials Specifications for Nuclear Service. As the representative of ASTM C-21 Committee, the Chairman of Subcommittee V attended meetings of the United States of America Standards Institute, especially Committee N11, "Basic Materials and Materials Testing for Nuclear Service"; a new committee sponsored by ASTM. At an organizational meeting, the Chairman of Subcommittee V was elected a Vice-Chairman of N11. Other activities

TABLE IV

ASTM SUBCOMMITTEE V (C-21), "NUCLEAR APPLICATIONS"

TASK GROUPS

I. Task Group Plutonium (Metal)	(al)	II. Pluto	Tas oniu	2 E	II. Task Group Plutonium Dioxide	III. Tests	Tas	3 k	III. Task Group Tests
Chairman: C. C	C. Caldwell	Chairman: H. J. Anderson	н.	ч.	Anderson	Chairman: L. T. Corbin	г.	÷	Corbin
Members : D. B	D. Bristol	Members :	J.	Ŀ.	J. L. Brennan	Members :		р.	D. Bristol
H. R. Diot	Diot			D.	D. Bristol		J.	г.	J. L. Brennan
R. H. Gale	Jale		. М	В.	W. R. DeHollander		В.	н.	R. H. Gale
W. R. Jacoby	Jacoby		В.	M	R. M. Fulrath		J.	Μ	J. M. Kerr
J. A. Leary	leary		J.	н.	J. H. Handwerk		. М	ŗ.	W. L. Lyon
F. J. Miner	1iner		J.	.Μ	J. M. Kerr		Л.	D.	J. D. McClelland
W. M. Pardue	ardue		••	Ľ.	0. L. Kruger		м.	Μ.	M. Pardue
			J.	Α.	J. A. Leary		J.	Еч	Soppet
			. М	г.	W. L. Lyon		ς. Ω	D.	S. D. Stoddard
			J.	D.	J. D. McClelland				
			• تم	ч.	F. J. Miner				
			л.	آتا	F. Soppet				

ASTM-Nuclear Fuels

#### Anderson

include correspondence, telephone contacts and review of minutes from related ASTM Committees, including E-10, and Subcommittee 20.20, D-9/D-20.

These studies and liaison activities with other related committees have indicated that ceramic materials that are used in nuclear applications have expanded rapidly. Many of these materials have developed to the point where meaningful standards can be more fully developed and proposed to industry and government.

## PROPOSED STANDARDS

As mentioned, there is great interest by both government and industry for new and additional standards. For example, Table V shows several standards that are proposed, or were proposed or active in the former ASA N5.1 Committee. As you will notice these standards cover a broad area of nuclear ceramics. The need has been expressed by industry or government for these standards. There is not enough time and manpower to complete work on these standards in the present organization.

## PROPOSED ASTM C-26 COMMITTEE

In 1967, the Nuclear Standard Board of the USA Standards Institute assigned ASTM as sponsor of the NBS project N11 on Basic Materials and Materials Testing involved in Nuclear Service. To better implement their responsibility in the areas of Nuclear Fuel Materials, the ASTM Board of Directors authorized an expansion of scope and full committee status for the ASTM connected work on fuel materials.

As part of this expansion Subcommittee V (C-21) solicited and now includes members of the former ASA N5.1 Committee and several new members of industry. Thus, Subcommittee V is now a nucleus for the new proposed full Committee to be known as ASTM C-26.

A Steering Group was formed and a meeting was held recently to outline a proposed scope, bylaws, slate of officers, and to set an organization meeting for the new ASTM C-26 Committee.

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# ASTM SUBCOMMITTEE V (C-21), "NUCLEAR APPLICATIONS"

Status Remarks	ive Proposed project.	ive Draft.	<pre>ive Prepared by ANS, sponsored by N5-1 for ASA adoption, resolved and transmitted to NBS for approval (1965).</pre>	ctive Sixth draft prepared.	Inactive First draft.	Inactive	Inactive	Inactive		ive Under preparation.	Inactive Proposed project.
Ste	ar Active	Active	Active	ole Inac	Inac	Inac		Inac		Active	Inac
Standard Activities	<sup>o</sup> High Cross Section Materials for Nuclear Poisoning	° Uranium Dioxide Fuel Pellets	Plate-type Uranium-Aluminum Fuel	° Nuclear Grade Thorium Dioxide, Sinterable Inactive	Nuclear Grade Thorium Dioxide, Com- pactible	Referee Methods for the Chemical Analysis of Thorium-Bearing Fuels	Referee Methods of Analysis for Thorium Compounds	<sup>o</sup> Plutonium Standard for Referee Methods of Analysis for Plutonium Compounds	<ul> <li>Nuclear Grade Uranium Dioxide- Plutonium Dioxide Fuel Mixture</li> </ul>	° Nuclear Grade Uranium Carbide	° Standardization in the Milling and

ASTM-Nuclear Fuels

The title is:

Fuel, Control, and Moderator Materials for Nuclear Reactor Applications.

The scope is:

The promotion of knowledge and the development of methods of test, definitions of terms, classifications, and specifications for fissionable and fertile materials and solid control and moderator materials, excluding graphite, intended for use in nuclear core components. The activities will be coordinated with those of other ASTM Committees and other organizations.

Recommended Organization and Slate of Officers:

The steering group proposed that the officers consist of a chairman, a vice-chairman for fuels, a vice-chairman for control materials and a secretary. It is proposed that initially the committee would support five subcommittees.

- I. Executive
- II. Fuel and Fertile Materials
- III. Control Materials
  - IV. Moderator Materials
    - V. Methods of Test

It is proposed that the Executive Subcommittee would consist of the officers, the chairman of the other subcommittees and two members-at-large representing the utilities interest.

Candidates were proposed for the following offices (subject to the approval of their participation by their organizations).

> Chairman: H. J. Anderson (BNW) Vice-Chairman: (fuels) C. Caldwell (NUMEC) Vice-Chairman: (control)

L. T. Corbin (ORNL) is proposed as Chairman of Sub V on Methods of Test, and C. Caldwell as Chairman of Sub II on Fuel and Fertile Materials. D. Rhodes, F. Forscher, and H. Anderson are designated to propose candidates who could serve as (1) Secretary of the new committee, (2) Chairman of Sub III Control Materials, and (3) Chairman of Sub IV Moderator Materials.

Time and Place for Organization Meeting of New Committee:

The organization meeting of the proposed C26 Committee is scheduled for Tuesday, November 12, 1968 as part of the meeting of the American Nuclear Society in Washington, D. C. at 7:30 p.m. in the Franklin Room of the Sheraton Park Hotel.

Invitees will be asked to ratify the scope, organization, and by-laws of the new committee, and indicate their probable areas of participation. For further information, contact me or telephone Jim A. Dwyer, ASTM Headquarters (215-569-4200), Philadelphia, Pa.

#### CONCLUSION

In conclusion, Subcommittee V is active, but there is much work to be done. With the formation of a full ASTM Committee, the manpower will become available to complete and issue the standards required by industry and government.

# A REVIEW OF THE WORK OF ASTM SUBCOMMITTEE II, D9/D20, EFFECTS OF HIGH ENERGY RADIATION ON PLASTICS AND ELECTRICAL INSULATION

#### 0. Sisman

This subcommittee (recently renamed 20.20) is sponsored jointly by ASTM Committee D-9 on Electrical Insulating Materials, and ASTM Committee D-20 on Plastics. It is one of the older of the committees dealing with radiation effects. For several years after its conception, under Dr. D. S. Ballantine, much of the activities of the subcommittee were educational (symposia) and involved trying to determine just what was needed. We now participate in symposia essentially only in cooperation with ASTM Committee E-10. Our present organization is shown in Figure 1. The scopes of the sections are shown in Figure 2.

An exposure method -- D1672-66, Exposure of Polymeric Materials to High-Energy Radiation -- was approved as an ASTM tentative in 1961 and advanced to standard in 1966. Section A, which developed this standard, is now becoming very active in determining the need for and developing methods for specifications for plastics and electrical insulating materials to be used in a radiation field. Along with this, and perhaps even before the specifications, we will study methods of testing for radiation damage.

CHAIRMAN:	Oscar Sism	nan	SECRETARY:	W. W. Parl	kinson
	Oak Ridge	Nat'l.I	lab.	Oak Ridge	Nat'l.Lab.
LIAISON OF	FICER WITH	E-10:	Kent C. Humph	erys	
			E. G. and G.	Corporation	n
SECTION A	- EXPOSURE	METHOD			
Acting C	hairman: W	1. W. Pa	arkinson		
	C	)ak Ridg	ge National La	boratory	

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ASTM-High Energy Radiation
SECTION B - GAMMA RAY DOSIMETRY
  Chairman: Francis X. Rizzo
            Brookhaven National Laboratory
  Co-Chairman: Robert D. Jarrett, Sr.
                U.S.A. Natick Laboratories
SECTION C - BETA PARTICLES AND ACCELERATED ELECTRONS
  Chairman: Donald E. Smith
            High Voltage Engineering Corporation
SECTION D - NEUTRON DOSIMETRY
  Chairman: Kent C. Humpherys
             E. G. and G. Corporation
SECTION E - GAMMA RAY DOSIMETRY IN REACTOR IRRADIATIONS
  Chairman:
            Kent C. Humpherys
            E. G. and G. Corporation
  Co-Chairman: E. D. McGarry
                Harry Diamond Laboratory
SECTION F - CORRELATION OF ABSORBED AND EXPOSURE DOSES
  Chairman: Jerome Weiss
             Brookhaven National Laboratory
SECTION G - ELECTRICAL MEASUREMENTS DURING IRRADIATION
  Chairman: Robert S. Shane
             General Electric Company
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Figure 1. Organization of ASTM Joint Subcommittee II, D-9 and D-20 Effects of Nuclear and High-Energy Radiation.

D-20.20 (II) Effects of Nuclear High Energy Radiation SCOPE: Test methods for measuring the effects of exposure to nuclear high energy radiation, and specifications for plastics and electrical insulating materials to be used in a radiation field.

#### Sisman

# Section A on Exposure Methods

Procedures for irradiating plastics and electrical insulating materials and specifications for plastics and electrical insulating materials to be used in a radiation field. Section B on Gamma Ray Dosimetry

Procedures for determining the energy absorbed by a specimen from a field of gamma radiation. Section C on Beta Particle and Accelerated Electron Dosimetry

Procedures for determining the energy absorbed by a specimen from a beam of accelerated electrons or beta particles.

Section D on Neutron Dosimetry

Procedures for determining the energy absorbed by a specimen from a flux of neutrons.

Section E on Gamma Ray Dosimetry in Reactor Irradiations

Procedures for discriminating neutron dose from gamma ray dose in reactor irradiations.

Section F on Correlation of Absorbed and Exposure Doses

Procedures for converting the dose absorbed in a standard dosimeter to the dose absorbed by a specimen. Section G on Electrical Measurements During Irradiation

Procedures for determining the electrical properties of a specimen during and after exposure to a field of radiation.

Figure 2. Scopes of Subcommittee D-20.20 and its Sections.

It became obvious very soon after this subcommittee was organized that we could do very little until we had some standard dosimetry procedures. Sections B, C, D, E, and F are concerned with this problem. As it turns out, we are the only active committee currently working on gamma-ray and chargedparticle dosimetry, and for that reason it has become a major portion of our work. We have also, for that reason, attracted members from fields not closely related to plastics or insulating materials; e.g., food irradiation, sterilization, irradiation services, etc.

## ASTM-High Energy Radiation

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A standard procedure for the Fricke dosimeter -- D1671-63, Test for Absorbed Gamma Radiation Dose in the Fricke Dosimeter -- was approved in 1963. At present Sections B and C are working on a large number of dosimetry systems to see which are adaptable to standard procedures. These are all widely used systems, and are listed in Table 1. Each dosimetry system has been assigned to a task group chairman (also listed in Table 1), and we hope that many of these methods will be ready for round-robin testing quite soon.

In Section D we have written a procedure for determining: the energy absorbed in a specimen from fast neutrons. This is -- D2365-65T, Method of Calculation of Neutron Dose to Polymeric Materials and Application of Threshold-Foil Measurements. It has recently been advanced to standard. The methods of measuring the fast neutron flux by the use of threshold foils are being developed in ASTM Committee E-10. We will now devote more effort to methods for measuring the gamma dose in mixed neutron and gamma fields. This will be done in Section E.

Dosimeter System	Task Group Chairman
Ferrous-Cupric Sulfate	Robert Jarrett
	U.S.A. Natick Laboratories
	Ari Brynjolfsson
	U.S.A. Natick Laboratories
Perspex, Lucite	Francis X. Rizzo
	Brookhaven Nat'l Laboratory
Ceric Sulfate	S. I. Taimuty
	Stanford Research Institute
Oxalic Acid	Neils Holm
	RISO, Danish A.E.C.
Polyvinylchloride	Robert Jarrett
	U.S.A. Natick Laboratories

Table 1. Assignment of Responsibilities for ASTM Dosimetry Test Methods and Recommended Practices

S:	is	m	an
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Film Dye	William McLaughlin
	National Bureau of Standards
Calorimetry	Robert B. Oswald
	Harry Diamond Labs
Gas Phase	James Sutherland
	Brookhaven Nat'l Laboratory
De-aerated Ferrous Sulfate	Francis X. Rizzo
	Brookhaven Nat'l Laboratory
Aerated Ferrous Sulfate	Neils Holm
	RISO, Danish A.E.C.
Thermoluminescent	Jeff Webb
Lithium Fluoride	Isotopes, Inc.
Calcium Fluoride	
Lithium Borate	Cal Rainbolt
	E.G. and G.
Jlass	Klaus Becker
	Oak Ridge Nat'l Laboratory
Dosimeter Selection	Francis X. Rizzo
Criteria	Brookhaven Nat'l Laboratory

A method for converting the radiation dose in a dosineter to that in the material of interest has been developed in Section F. This tentative method -- D2568-66T, Correlation of Absorbed and Exposure Dose -- has recently been advanced to standard. This method has been rather oversimplified because it was written to apply to plastics. It is limited to elements with mass up to that of chlorine, and to gamma-ray energies between .5 and 3 MeV. With caution one can use this procedure for x-rays and electrons, and a little outside these limits. However, a more complicated procedure will be required for broader coverage.

Our last section, G, is an important one, but not an Pasy area in which to produce standard methods. Much work has been done here in determining just what is needed. The meetings are being held in conjunction with meetings of E-10, Sub.VI, Sections E and F. Much of the interest here is presently for space application.



# ASTM, COMMITTEE D19, METHODS FOR RADIOCHEMICAL ANALYSIS OF WATER D. L. Reid

Formal organization of a Task Group in Committee D19 to sponsor analytical radiochemical methods for the field of Tracer technology was started early in 1956 with the first scoping meeting in June of that year. Word of this new ASTM activity spread quite rapidly and prior to publication of the first Measurement method suggestions from industry quickly broadened the scope of the group to the all inclusive radiochemical analysis of industrial water and industrial waste water. Growth in the membership and the quantity of work created a subcommittee in 1959 comprised of four basic Task Groups. The scope of the task groups, unlike other ASTM Task Groups, covered broad categories of radioactive elements rather than a single element and encompassed all radionuclides according to production or origin namely, Fission Products, Reactor Cooling Water Contamination, Naturally Occurring Radionuclides and Measurement of Radioactivity. Task Groups on Uranium, Heavy Water, Tritium, and Evaluation of Methods were added as required. This organization broadened the participation of each member since most were members of all task groups and allowed processing of several methods concurrently with a small number of members. It also created a full three-day schedule of meetings for most members.

The one disadvantage to this universal participation type organization is in the quantity of work to be sandwiched into each member's business schedule. However, this one disadvantage is apparently offset by the gain from the crosspollination of ideas and comments which adds uniformity to the methods and has a tendency to reduce the total time required to prepare a method for publication. We now vacillate between a 3 and 4 day meeting, dependent upon the projected agenda, twice a year.

#### ASTM-Water

A recent Committee reorganization elevated the Task Groups to Sections since the Task Group title was reserved for groups working on a single method. At the same time, some name changes were in order to broaden the scope of two sections. The subcommittee now has five sections - as shown in Table I. The scope of the subcommittee is the preparation of standards for physical and chemical testing of water and water formed deposits where radioactivity or radiochemical properties are controlling.

#### TABLE I

ASTM COMMITTEE D19

SUBCOMMITTEE 19.4

METHODS OF RADIOCHEMICAL ANALYSIS OF WATER

Chairman - D. L. Reid

Vice Chairman - S. A.	Reynolds					
Sections	<u>Chairmen</u>					
Measurement of Radioactivity	B'. Kahn					
Fission Products J. A. Corbett						
Heavy Radionuclides	G. A. Welford					
Activation Products	J. A. Martucci					
Evaluation of Methods	S. L. Williams					

The twenty-three members represent laboratories of the AEC, AEC Contractors, National Bureau of Standards, U. S. Public Health, Power Reactor Manufacturers, and Power Reactor Owners. This complement gives us an excellent experience cross section ranging from low level to high level analytical radiochemistry. Their personal experience in the field ranges from 5 to 20 years.

Nineteen methods have been published of which eight are standards, four are in the process of being advanced to standards and seven are tentatives. Ten additional methods are in various stages of development. The methods are written for use by trained technicians, not chemists, which adds considerably to the detail of a method, but releases the radiochemist for development work and trouble shooting.

Since most industrial water returns to the environs, and disposal of radioactive pollutants must be closely controlled, priorities for producing methods were assigned on the combined factors of biological significance, maximum permissible concentrations and probability of occurrence in water. With most of the methods for the major biologically significant isotopes completed, the predominant nuclides in various categories now dictates the order. Of course, the concise lucid document - unless, of course, you make it a full-time job for those involved.

Two major problems most frequently encountered, other than keeping our testing of the methods abreast of publication, are meeting the deadlines we establish and lack of a detailed review for each draft produced. The reason for the first is quite obvious - we are basically optimists or can't predict what the boss has in store for us. The other is apparently related to familiarity, with the thoroughness of the review decreasing with each successive issue. We too readily assume that if it was once correct, it will ever remain so in subsequent drafts and the tendency is to concentrate our thoughts on the sections which were revised. For example, sometime between the first draft and the published document a decimal point was omitted from a 2.0 milliliter reagent volume and as you might suspect, the procedure fell apart at this point. Fortunately, testing of the method caught the error and the necessary correction was made. Other errors not affecting the outcome of the analysis are best caught by detailed reviews of galley proofs. This requires 2 or better 3 dedicated members bent on producing letter-perfect documents.

In the early part of the program, the variation in counting instruments between laboratories throughout the country, each with its own characteristics and peculiarities, forced a generalization of the measurement sections for a particular radionuclude rather than limiting the procedure to the best counting method. For example, if the isotope

Reid

#### ASTM-Water

could be more accurately determined by gamma counting, but could also be measured by beta counters, both methods were suggested in an effort not to exclude even one laboratory from using the method. Similar considerations had to be applied for the types of counters in a specific category such as end-window,  $2 \pi$  gas flow and  $2 \pi$  internal beta counters. This had a tendency to dilute the positives and eliminate the implied negatives used as guides in analytical methods.

Fortunately, the rapid improvement in all aspects of analytical radiochemistry and more specifically the advancements in instrumentation are responsible for increasing first methods attacked were those for measurement of the radioactivity of the samples.

The procedure leading to publication of a method is rather simple when reduced to writing, but the labor and time involved is something else. Favorite methods for the radionuclide of interest are submitted by the members for study and discussion at the next meeting. After agreement is reached on the best test method, a "volunteer" prepares the first draft which is circulated for review and comments prior to and discussion at the next meeting. Successive drafts are processed in the same manner until the method is approved by the subcommittee.

The method is then tested for clarity and precision by a trained technician from a lab other than the major author of the method. Regardless of the experience of the technician, they are given instructions to follow the method as written not as they think it should be run based on their experience. It didn't take too long to find out that the written word is not interpreted the same by all and that extreme care is required to accurately convey to each prospective user exactly what was intended. If no questions are posed by the technicians and precision and accuracy are as expected, the method is recommended for publication in the Preprinted Report.

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#### Reid

All methods or revision to methods appearing in this publication are subject to letter ballot approval of the entire Committee. Attempts made to resolve all supported negative votes are documented. A 10% negative vote forestalls publication of the method in the Book of Standards. If approved, round-robin testing of the method is in order and again if successful, the method can be advanced to standard status - a very simple statement for a complex problem.

The length of time and the number of drafts from conception to publication is related to the complexity of the method and has varied from 1-1/2 to 3 years and 2 to 5 drafts. This may seem excessive to those not familiar with the process of writing a standard method or specification acceptable to all, but is probably average or better than average and apparently is required to produce an errorless the nationwide uniformity in counting equipment and the methods can be made more specific. Just one round-robin test vill readily convince you that this is an essential requirenent of a standard radiochemical analytical method. Consequently, the problem is diminishing with increasing age and nethods can be more specific and are somewhat easier to proluce as well as to interpret.

The length of time required to hammer out a method seems interminable and it is a great relief when the method is finally advanced to standard. But the changing technology and instrumentation of radiochemistry will not allow you to rest on your laurels. If your goal is to provide the best standards for the existing state-of-the-art, then a periodic review of each method is essential.

This is built into the ASTM system which requires a review of all standard methods at least every five years. In radiochemistry we found this to be too infrequent for some methods and changes in the procedures were and are made as the technology dictates. The yearly ASTM publication of the Book of Standards provides a procedure for altering the method each year if substance changes are required to keep abreast of the advancing technology.

#### ASTM-Water

There is much time, effort, and expense accrued in producing a Standard Method of analysis and duplication by two or more societies or organizations creates a flagrant waste of time, manpower, and money. Money we may have enough of, but from our experience we have very little time we can afford to waste in replowing the ground. Although there may be too many established barriers, prejudices and jealousies for societies or organizations to unite their "Standards" efforts in most fields, it is not too late for radiochemists of all Societies and Standards Organizations to initiate a cooperative effort. Some duplication of effort in sponsoring radiochemical methods has already occurred and a united effort for the good of Society is past due. Analytical radiochemistry has two built-in characteristics which provide a common ground on which all organizations can meet. Except for direct counting of a solid, liquid or gaseous sample, all samples at some time are in a liquid state and all must be counted by a particular type of instrument having specific characteristics. Consequently, the basic analytical radiochemical method, even for direct counting of the sample, can be applied by all physical sciences. If in fact this common ground does exist and is not just a mirage, is there any sound legitimate reason against uniting all analytical radiochemists regardless of their Society heritage to eliminate the waste compounded by replication of analytical standards? Such a united organization would also increase the impact of the U. S. standard methods on the international scene.

It is true that pretreatment of the sample may differ widely but this is no deterrent to producing a basic method. Once that basic method is established, the Society or organization can insert specialized pretreatments dictated by the type of sample to obtain the chemical or physical state detailed in the first step of the basic method. How this united effort can be accomplished is beyond the scope of this discussion and my knowledge of the machinations of the various standards - writing bodies. Those of you more fa-

#### Reid

miliar with this protocol can probably find a simple solution. Possibly USASI is a good starting point since its role is that of a clearing house and coordinator for national standards activities. Any suggestions or comments favorable or otherwise, either written or oral would be welcome. Possibly the subject is suitable for discussion during tomorrow's sessions if time permits.

Subcommittee IX of Committee D19 has just scratched the surface. A much larger committee would accelerate the process immeasurably and possibly the issue of approved Standard Methods for analytical radiochemistry might catch up with the need.



# ASTM COMMITTEE E-10 ON RADIOISOTOPES AND RADIATION EFFECTS

### Duane N. Sunderman

ASTM Committee E-10 was organized in 1951 in the early days of industrial involvement in atomic energy. As an "E" committee, its primary mission was to provide an advisory service to other ASTM committees in the radioisotope applications and radiation effects areas. It has also evolved areas of standards development where Committee E-10 has taken the initiative and has had principal concern.

The committee's mode of operation is characteristic of ASTM groups, that of voluntary standardization based upon the consensus principle, involving all interested and affected parties. Total membership, including the main and subcommitees, is about 200. Two or three meetings of 2 or 3 days duration are held each year and, since its organization, some 25 standard procedures and recommended practices have been developed. A second activity of the committee concerns the dissemination of information on radioisotope applications and radiation effects within the ASTM setting. Symposia have been conducted in all areas of the committee's interest. For the past 6 or 8 years, such symposia have been conducted at a rate somewhat higher than one per year. Liaison is also an important activity of Committee E-10 since it is not only concerned that its efforts not duplicate or be duplicated by other standards groups, but also that all areas of its interest be covered either within or outside the ASTM framework. Current liaison activities include about one-half of the USASI "N" committees; related ASTM groups such as D9/D20, D-19, and E-21; goverment agencies including USAEC, DASA, NACA, and NBS; and standards groups within professional societies such as ANS and ASME. These liaison activities include cross-membership and formal reports of the activities of the other groups at subcommittee and E-10 main committee meetings. 101

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The organizational structure of ASTM Committee E-10 is presented in Table 1. Our two Vice Chairmen assist the Chairman by each working closely with a group of subcommittees in planning and carrying out their programs. The Liaison Officer coordinates all liaison activities, assuring that these positions are properly staffed and that the individual liaison responsibilities are properly carried out.

TABLE 1.

ASTM COMMITTEE E-10 ON RADIOISOTOPES AND RADIATION EFFECTS

CHAIRMAN	- D. N.	SUNDERMAN				
VICE CHAIRMAN	- L. E.	STEELE, M. M. TURKANIS				
SECRETARY	- R. L.	STUART				
LIAISON	- A. L.	BEMENT				
SUBCOMMITTEES						

1.	NUCLEAR FUEL BURNUP	-	R.(	C. SHANK
2.	RADIATION INDUCED CHANGES IN METALS	-	J.	MOTEFF
3.	TRACER APPLICATIONS	-	E.H	E. WICKER
4.	MEASUREMENT USING EXTERNAL RADIATION SOURCES	-	W.	GUNDERMAN
5.	DOSIMETRY	-	R.	H. LEWIS
6.	SPACE RADIATION EFFECTS	-	Ј.	ROMANKO

The standards development activities are conducted in six subcommittees, each operating within a well defined scope. Subcommittee I on Nuclear Fuel Burnup is chaired by Ralph Shank of Idaho Nuclear, who is present and will describe its activities in a later paper.

Subcommittee II on Radiation Induced Changes in Metals, chaired by John Moteff of General Electric Company, will be described by Arden Bement who was until recently its Chairman.

Subcommittee III on Tracer Applications is chaired by E. E. Wicker of United States Steel. Early in the activities of this group, they were concerned with the development of

#### Sunderman

standard procedures for determining the radiochemical purity of pure radioisotope preparations. Two standards developed at that time are E-181, a General Analysis of Radioisotopes, and E-182, the Analysis of Phosphorus-32. They are currently busy in the activation analysis area, just completing a procedure for the measurement of oxygen by 14 Mev neutron activation. They will be expanding these activities in the next few months and would welcome the addition of interested parties to their membership roles.

Subcommittee IV on Measurement Using External Radiation Sources is chaired by Bill Gunderman of the Highway Research Board. They are heavily involved with other ASTM groups in the preparation of standard procedures for the measurement of moisture and density by radiation techniques. This group is also preparing a procedure for the calibration of such instruments.

Subcommittee V on Dosimetry is chaired by Bob Lewis of Babcock and Wilcox. Early efforts of this group concerned nomenclature involving close coordination with ISO in the preparation of glossaries of nuclear terms. Dosimetry of beta and gamma radiation, while also an early activity of this group, was discontinued when it was picked up by the ASTM joint D-9/D-20 committee in the late 1950's. Neutron dosimetry continues to be the major activity of Subcommittee V. From this work has come a series of six standard procedures (E-261, 262, 263, 264, 265, and 266) covering thermal and fast neutron dosimetry by activation techniques using cobalt, nickel, iron, sulfur, and aluminum. These procedures are currently undergoing evaluation by round robin analyses. Procedures are in preparation for the use of fission foils. This group is also studying the use of advanced computational techniques for developing neutron spectra and dose from activation data.

Subcommittee VI on Space Radiation is chaired by John Romanko of General Dynamics--Fort Worth. Liaison with ASTM Committee E-21 on Space Simulation is particularly important

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to this group because of their closely related missions. For standards development, this subcommittee is further divided into sections on space radiation environment, indigeneous space radiation effects, vacuum-radiation effects, temperature-radiation effects, electronic components and modules, and pulsed radiation effects. Standard procedures and recommended practices are being prepared in a number of these areas.

In conclusion, an effort has been made in Committee E-10 to establish a framework of organization and operational procedures sensitive and responsive to the needs of ASTM, industry, and government in the nuclear areas. This includes the conduct of liaison, informational, and standardization activities in a variety of areas within the limits of our competence.

# ACTIVITIES OF ASTM: E-10; SUBCOMMITTEE I ON NUCLEAR FUEL BURNUP

## R. C. Shank

The ASTM E-10 Subcommittee I on Nuclear Fuel Burnup is composed of the following members:

Interest	Members	Observers
Reactor Designers & Fuel	12	6
Fabricators		
Power Reactor Operators	l	0
Fuel Reprocessors	4	0
General Interest	10	5
Total	27	11

It is difficult to categorize many of the members because some of them have interest in more than one field, but I have tried to put them in the field of their major interest. Many of them are either partially or wholly supported by AEC but not all. As you can see, there is a shortage of members from the Power Reactor Industry. This is unfortunate because the Reactor Operators have a big stake in this work. This is one of the few times that considerable work has been done toward developing standard methods to solve a problem before it arises, rather than after, and they should be participating in it. I anticipate a greater interest will be taken by these people after there has been a disagreement someplace in the fuel cycle. I think AEC should be congratulated in encouraging this work before the fact.

The burnup work was started in 1956 as a Group in the Dosimetry Task Force of the Radiation Effects Subcommittee II. To have any meaning, the effect of radiation must be evaluated against a known amount of radiation, hence the Dosimetry Task Force. It also seemed reasonable that while the irradiation dose was being measured, it would be well to know the amount of burnup that had occurred in the case of fuel material, so the Burnup Group was organized. At that time the job was sim-105

### ASTM-Fuel Burnup

ple. The only requirements were a method to analyze for uranium and one for cesium-137, from which burnup could be easily calculated. That was during the time of relatively short That is still all that is burnups and at low temperatures. needed at those conditions. But the real need is no longer at those conditions. Power reactors are being operated for many thousands of megawatt days and at temperatures so high that cesium becomes mobile in the fuel rods. The burnup group is attempting to standardize methods that will give accurate results under the new conditions. This is not an easy task. Our efforts are restricted to power reactor problems and no attempt is being made to solve all burnup problems, especially 11 those of strictly a development nature. As a result of the increased work, the group became a full subcommittee during the reorganization of Committee E-10 in 1966.

The following scope of the Subcommittee was developed at that time:

#### SCOPE

"The Nuclear Fuel Burnup Subcommittee shall provide advice and counsel to other ASTM Committees concerning all matters of determining burnup of nuclear fuel. It shall also write ASTM standard methods for the measurement of burnup."

The following methods have been prepared by the subcommittee:

E-217-63T	Tentative Method for Determination of Ura-
	nium by Controlled-Potential Coulometry.
E-267-65T	Tentative Method for Determination of Ura-
	nium and Plutonium in Aqueous Solutions.
E-318-67T	Tentative Method for Colorimetric Determina-
	tion of Uranium in Aqueous Solutions.
E-320-67T	Tentative Methods for Radiochemical Determi-
	nation of Cesium-137 in Nuclear Fuel Solu-
	tions.
E-219-63T	Tentative Method of Test for Atom Per Cent
	Fission in Uranium Fuel (Radiochemical Meth-
	od).

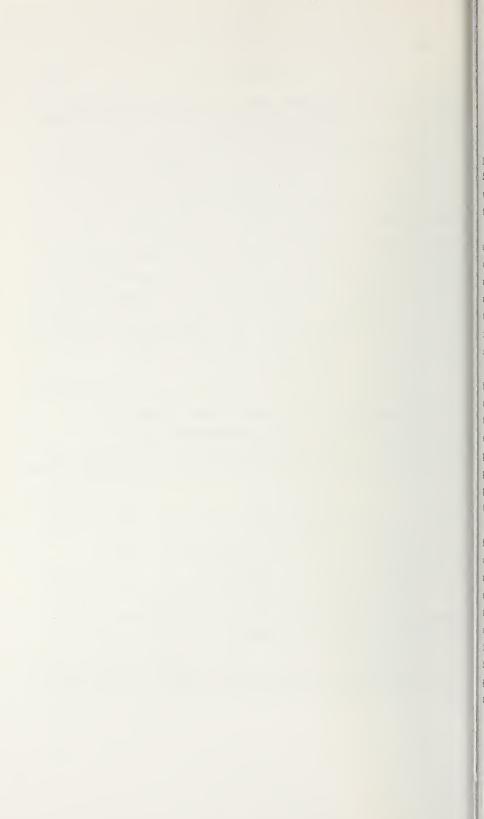
- E-244-65T Tentative Method of Test for Atom Per Cent Fission in Uranium and Plutonium Fuel (Mass Spectrometric Method).
- E-321-67T Tentative Method for Determination of Atom Per Cent Fission in Uranium and Plutonium Fuel (Stable Fission Product Nd-148 Method).

All of the methods except the U-Pu Concentration and Isotopic Abundances (E-267) and the Cs-137 Methods (E-320) have cleared the Subcommittee to become standards. They are waiting to be balloted by Committee E-10. Method E-267 is cleared as far as uranium is concerned but the plutonium portion has not been round-robin tested. The cesium method covers two procedures, (1) one by cesium perchlorate, and (2) the other by chloroplatinate. The chloroplatinate method is satisfactory but efforts are being made to improve the precision of the perchloric acid method.

Although each method describes how to make the necessary standards, the precision of the methods could be improved if standard materials were available commercially. The materials needed and the form of standardization are:

Material	Form of Standardization	<u>Availability</u>		
Cs-137	Atoms/ampoule	Nuclear Chicago Cs-137-ASTM		
Nd-150	Atoms/ampoule	Not available		
U-233	Atoms/ampoule	Not available		
Pu-242	Atoms/ampoule	Not available		
U-235/U-238	Series of Known Ratios	NBS		
U-235/U-236	Series of Known Ratios	Not available		
Pu-239/Pu-240	Series of Known Ratios	Not available		

This discussion has covered the work that has been completed to date in useable form by the Subcommittee on Nuclear Fuel Burnup. Work is in progress on methods for some other fission products which can be used as burnup monitors. All the work, so far, has been for thermal reactors. Work on fast reactor burnup will be started as information becomes available.



# GOVERNMENT ASSISTANCE IN DEVELOPING VOLUNTARY STANDARDS D. R. Mackay

It is a pleasure to have this opportunity to speak to you today about the Commerce Department's voluntary Product Standards program. The Office of Engineering Standards Services of the National Bureau of Standards is responsible for this program.

This morning and so far this afternoon we have heard representatives of a number of private standards groups describe and explain their activities. You may be wondering why, with all these private groups, a government program exists and where it fits in the national standards structure. In addition to answering these questions, I would like to give you an idea of our function and how we assist in the formation of standards.

Let me begin by mentioning the requirements which must be met before the Department participates in the development of a standard. First, the proposed standard must not be contrary to the public interest. In this requirement are two essential words which are the key to the purpose of our program--the words are "public interest." The government's program is first and foremost a service to the public, to the producers of the product standardized, as well as to the distributors and users of the product.

Secondly, a proposed standard, to be considered, must have national effect or implication. Our program is not concerned with local or regional problems. Thirdly, a standard must have apparent industry-wide interest or endorsement; otherwise, it might be foolish to initiate the development of a standard. And finally, the standard must be such that it cannot be processed according to the <u>needs</u> or <u>desires</u> of the industry by a nationally recognized private standardizing body. In other words, we are not in competition with private groups, instead we exist to complement their activities, and serve the public interest.

## Government-NBS Voluntary Standards

The government's voluntary standards activities began during World War 1. At that time industry-government cooperation was essential to the war effort. The Conservation Division of the War Industries Board was created to see that the largest possible amounts of labor, capital, materials and equipment were released for the war effort. The governmentindustry program was established to conserve materials and eliminate waste through standardization and simplification of varieties and sizes of commonly used mass-produced items.

But when the war ended so did compulsory standardization and many manufacturers quickly returned to the old uneconomic conditions of over-variety. The situation was aggravated in 1921 when a delayed post-war depression struck and manufacturers felt they must offer variety to obtain more sales. Herbert Hoover, as a prominent engineer and later as Secretary of Commerce, was one individual who was so concerned that he sought to rid industry of waste through the establishment of standardization programs.

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In 1921, while he was President of the American Engineering Societies, Hoover appointed a committee to study the then existing conditions of waste in industry and to make suggestions as to possible remedies. The committee studied six typical industries and found that preventable waste of labor and materials averaged almost 50 percent in those industries. The committee's report entitled "Waste in Industry" estimated that 10 billion dollars a year--1921 dollars--could be saved through standardization and simplification alone.

The report suggested that the government should play an active part in the formation of industry standardization committees. When he became Secretary of Commerce, Herbert Hoover had the opportunity to implement this recommendation. He established within the Department of Commerce a Division of Simplified Practice. This Division played a major role in promoting the development of voluntary industry standards. Its publications entitled "Simplified Practice Recommendations" provided for the voluntary reduction in the number of

#### Mackay

sizes and varieties of many products. For a time it led a massive national drive for standardization. In 1927, the scope of the government's activities was broadened to include a Commercial Standards Unit which developed, cooperatively with industry groups, standards establishing quality requirements for specific products. Through the years the program has been assigned to different offices within the Department of Commerce, it has changed names several times, and it has experienced consolidation--the Simplified Practice Division and the Commercial Standards Division were united in one Commodity Standards Division within the Department of Commerce.

In 1963, a reorganization resulted in the work being transferred to the National Bureau of Standards. At this time it was decided that instead of two publications, Commercial Standards and Simplified Practice Recommendations, only one publication series would be issued to be called "Product Standards." These standards could include quality requirements as well as simplification practices. The one thing that has not changed with time is the goal of the program: to aid industry in the development of standards which are deemed to be in the public interest.

Our procedures, revised in December of 1965 and amended May of 1968, reflect the emphasis on this goal. I would like to summarize those procedures for you. The process begins when an interested group, whether made up of producers, distributors, consumers, users, testing laboratories, or a government agency asks the Bureau to participate in the development of a voluntary standard. The Bureau then determines if the request is feasible and if it conforms to the requirements I mentioned previously, including--is it in the public interest?

When the request is approved, a specific proposal is developed in consultation with interested trade groups and interested government agencies. This proposal is then subjected to an impartial technical review by an appropriate

### Government-NBS Voluntary Standards

Government agency or agencies interested in the standard. If it is appropriate, the technical review may be accomplished by an unbiased group outside of the Federal government. A draft of the proposal is then circulated for consideration and comment to interested groups including consumers and users.

At this point, a Standard Review Committee is established to review the amended draft which incorporates the suggestions received from any segment of the industry. The procedures specify that the Standard Review Committee must be representative of all groups interested in the product for which the standard is sought. It is also our policy to see that small business as well as big business is represented on the committee. Once the committee approves the proposal, it is distributed to all known producers and a representative sampling of distributors, users, consumers and general interest groups for final consideration and acceptance. Any objections received from these groups are carefully considered by NBS. If there are no significant objections and if the proposal is supported by a "consensus," the NBS announces the approval of the proposal as a Product Standard.

Finally, prior to the printing of a Product Standard, a Standing Committee is named to review the standard within five years of its issuance, to consider any proposals to revise or amend the standard and to provide such interpretations as may be required. This committee is essentially identical in composition to the Standard Review Committee as to membership and procedures.

A standard, then, is submitted twice to the general industry for consideration, once to a special committee made up of representatives from the interested groups and once to an impartial group for technical review. It should be noted that any individual or company is at liberty to comment during either distribution. Generally, a press release is issued when the recommended standard is distributed for acceptance, and in many instances, a release is issued when the proposed standard is distributed for initial comments.

#### Mackay

At this point, let me explain what is meant by "consensus." The latest amendment to our procedures established a specific definition of consensus in terms of the numerical percentages. It is now required that a standard be supported by at least 70 percent of those responding to the distribution of the recommended standard in the production segment, in the distributor segment, and in the user or consumer segment of the industry. Furthermore, the procedures require that the average percentage of acceptance for each of the three segments be not less than 75 percent. The amended procedures also provide a second definition for consensus which involves lower percentages. This alternative definition is implemented for standards which are considered to be in the public interest but which did not receive the percentages of acceptance previously mentioned. Under this second procedure, the minimum acceptability in any segment of the industry must be not less than 60 percent and the average of the three segments must be not less than 66-2/3 percent. This procedure also involves the holding of a public hearing to allow the Department to substantiate the importance of the standard to the public.

I would now like to enumerate the specific responsibilities of the National Bureau of Standards and of the group proposing the standard. The Department assists in the formation of a voluntary standard through the following: It acts as an unbiased coordinator in the development of the standard; it provides editorial assistance in the preparation of the standard; it supplies such assistance and review as is required to assure the technical soundness of the standard; it sees that the standard is representative of the views of producers, distributors, users and consumers; it seeks satisfactory adjustment of valid points of disagreement; and finally, it publishes the standard.

The group proposing the standard and the industry which is affected by it have the responsibility of: Initiating and participating in the development of a standard; providing

## Government-NBS Voluntary Standards

technical counsel on a standard; and promoting the use of, and support for the standard.

Our voluntary standards may cover definitions, classes, sizes, dimensions, capacities, quality levels, performance criteria, testing equipment, and test procedures. They may vary in scope from the most complex requirements for precision instruments to size standards for the simplest of items such as 2 x 4 lumber. At present, we do not have any Product Standards in the field of nuclear chemistry and technology; however, if a group came to us with special problems that could be solved through the development of voluntary standards, we could call on the vast resources of the NBS's Center for Radiation Research for assistance in developing such standards. The Center's personnel have been doing extremely important work in the nuclear field, particularly in the measurement and calibration area. The Center could provide excellent technical assistance in the development of standards. The Atomic Energy Commission and the Public Health Service could also assist in the process.

In closing, let me suggest that our procedures, our facilities, and our services are available to those groups which have problems which could be alleviated if not eliminated through the development of voluntary standards which are in the public interest.

# THE STANDARD REFERENCE MATERIAL PROGRAM OF THE NATIONAL BUREAU OF STANDARDS J. Paul Cali

The National Bureau of Standards (NBS) has been involved in the nuclear field from its inception in the early 1940's. Through the Standard Reference Materials (SRM) program, the Bureau has played a leading role in the issuance of SRM's directed toward the quality control of ores, metals, and fissile material used in many areas of nuclear applications, as well as the certification of a wide variety of radioisotopes used in the calibration and standardization of nuclear instrumentation.

It is the purpose of this paper to set forth the operations of the Office of Standard Reference Materials (OSRM), and to show the variety of SRM's now available for use in this field of science and technology.

A SRM is "a well-characterized material, produced in quanity, which calibrates a measurement system or produces meaningful scientific data". Several implications are inherent in this definition. "Well-characterized" at NBS implies that any certified value or property is determined by two or more independent methods of analysis, or possibly in wellestablished systems, by a method whose accuracy has been carefully assessed and whose systematic errors are small relative to the degree of accuracy required. SRM's are "materials" not methods nor specifications, although NBS-SRM's are often an integral part of a standard method. In general, they are homogeneous solids, although liquid and gas SRM's are issued where solids are not suitable, convenient, or available. For SRM's, issued as gases or liquids, particular attention is given to stability considerations. "Produced in quantity" is a necessary criterion so that all potential users may be assured of an adequate supply over a reasonable period of time. In general, a 10-year supply of stable SRM's is produced and certified at one time. This obviously cannot 115

#### Government-NBS SRM's

be the case of radioactive SRM's of short to medium half-life, nor for materials, such as cholesterol, whose shelf-life stability over a fairly long period is not known or wellestablished.

SRM's are produced and used for four basic purposes; 1) to facilitate the exchange of goods - e.g., the Sucrose SRM is used to calibrate polarimeters at Customs laboratories for levying duty on imported sugar; 2) to permit quality control - e.g., the quality of most of the steel produced in the U. S. is controlled through the use of emission or x-ray spectrometers which have been calibrated using NBS-SRM's; 3) to determine performance characteristics - e.g., metalloorganics in oil SRM's are used by the Department of Defense and the transportation industry to program maintenance schedules and to predict failure rates of engines; 4) and, to characterize at scientific frontiers - e.g., high-purity and doped platinum SRM's are used to calibrate spark-source mass spectrographs at the sub-ppm level.

The OSRM is responsible for both the technical and administrative functions of this program. Table 1 shows the functional organization of the OSRM. It should be pointed out that the actual measurement and certification process is carried out in the Technical Divisions of NBS.

Requests for new and renewal SRM's are, in general, received from many sources: other government agencies (ESSA, NIH, AEC, etc.); industry (steel, rubber, etc.); standardizing bodies (USASI, ASTM, ISO, etc.); and, often from individual scientists and engineers who recognize a specific need. However, because the resources allocated to this program are insufficient to produce all requested SRM's, the OSRM rates each request in order to establish a priority listing of all the requests received, or remaining from previous years. Some of the factors considered in this rating are: the availability of non-NBS-SRM's which could be used as an alternate source; whether NBS Technical Division resources are available, and if so, the status of the support-

## Table 1. Functional organization of OSRM.

- 1. Gather requests for new and renewal SRM's
- 2. Formal justification (7 points)
- 3. Establishes *priorities* on annual basis; establish *projects* for current SRM production
- 4. Allocates funds to operating divisions for current projects
- 5. Locates sources of supply for SRM's; procure test lots; evaluate for homogeneity and compliance with specifications. (Internal NBS testing by operating divisions through OSRM)
- 6. Coordinate technical measurements and certification. Coordinate cooperative analyses.
- 7. Prepare certificate of analysis; announcements of new and renewal SRM's; technical publicity; customer liaison
- 8. Establish prices and marketing policy
- 9. Inventory control, cost accounting, sales, distribution

ing research; the number of requests, the sales potential, and cost of production (this is a cost-recoverable program and some consideration therefore must be given to these factors); whether non-NBS support is available or likely in terms of direct financial support, gifts of materials, or cooperative analytical time; whether SRM's are widely accepted and used in the particular area or industry requesting an SRM; the availability and ease of procuring the basic material; and finally, the risk involved in terms of technically difficult measurement or materials problems.

Having established the priority list, the OSRM then sub-contracts to the supporting Technical Divisions of NBS those SRM projects which can be funded from the dollar resources made available to the program by the Director. The materials for these authorized projects are specified and procured by the technical coordinators of the OSRM. As the materials are received they are tested in the various Divisions of NBS for compliance with specifications and carefully measured for homogeneity of the entire lot.

### Government-NBS SRM's

At the present time thirteen Technical Divisions of NBS are measuring and certifying over 150 materials for such properties as chemical composition, radioactivity, viscosity, thermodynamic properties, color, metal coating thickness, and a host of other chemical and physical properties. This technical diversity is illustrated in the listing of table 2

Technical division	Examples of technical competences
Metrology	Thermal expansion; phase transitions; colorimetry; photo- grammetry
Mechanics	Pressure measurements; humidity; vacuum techniques
Heat	Thermodynamic properties measurements; calorimetry – heat of combustion, fusion, solution, etc.
Radio Standards Engineering	Dielectric properties, permittivity
Cryogenics	Residual resistivity ratio; low-temperature measurements
Analytical Chemistry	Trace element analysis; electrochemistry; microchemistry
Polymers	Molecular weight determination; dielectric properties
Metallurgy	Electrolysis; metal deposition; x-ray diffraction; quantitative metallography
Inorganic Materials	Crystallography; solid-state physics
Physical Chemistry	NMR; thermochemistry; mass spectrometry; radiation chemistry
Product Evaluation	Physical properties of rubbers (viscosity, stress-strain)
Building Research	Fire research; materials durability and analysis
Nuclear Radiation	Radioisotopes; nuclear properties
Applied Mathematics	Statistics; experimental design

Table 2.	Resources available for measurement and
	certification of Standard Reference Materials.

showing the Technical Divisions now involved in the SRM program and an example of some of the competences used in each Division.

Well over 450 SRM's are on the request list and would require over \$15,000,000 to complete. The present level of support is \$1,700,000 per year. This backlog is shown in table 3 broken down by national concern area, a categorization used within the Federal structure for planning and budgeting purposes.

Table 3. Standard Reference Materials backlog.

National concern area	Typical example	Typical requestor	Cost (\$K)
Defense and Aero- Space	Titanium alloys	DOD, Aero-Space Ind. Assoc.	4,631
Economic Growth	Plutonium	AEC, AISI, General Motors	3,756
Education and Technology	Polyethylene	ASTM, ACS, Plastics Industry	3,548
Consumer Interests, Safety, Government Operations	Test Charts, Document Control	Ford, FBI, Navy	1,388
Conservation, Resources Development	Fertilizer SRM's	USDA, Bureau Mines, Customs	905
Health, Pollution Control	SO <sub>2</sub> in Air Bilirubin	NIH, NCAPC, AACC	860
			A15 000

Total funds needed for 251 new SRM's

\$15,088

## Government-NBS SRM's

The present SRM inventory contains 665 items in 70 categories. Of these, there are now available to users in the nuclear field well over 75 SRM's of direct interest and application. Table 4 lists these, but does not include the wide variety of both ferrous and non-ferrous metals SRM's, except as shown, which have utility for the metals applications in this area of technology.

## Table 4. SRM's in inventory of interest to nuclear field.

General class	Number items available	Examples
Radioactivity SRM's:		
Alpha-Ray	2	polonium-210; americium-241
Beta; Gamma; E.C.	21	carbon-14; sodium-22; krypton-85
Radium/Radon	14	radium solution (radon analysis)
Special Nuclear Materials	18	plutonium metal; uranium-235 (graded series)
Isotopic Reference SRM's	9	chlorine; copper; lead (set of 3)
Refractories/Ceramics	8	glass; low boron; aluminum oxide
Ores	1	uranium ore
Metals <sup>a</sup>	4	zircaloy-2F
Mössbauer	1	sodium nitroprusside crystal

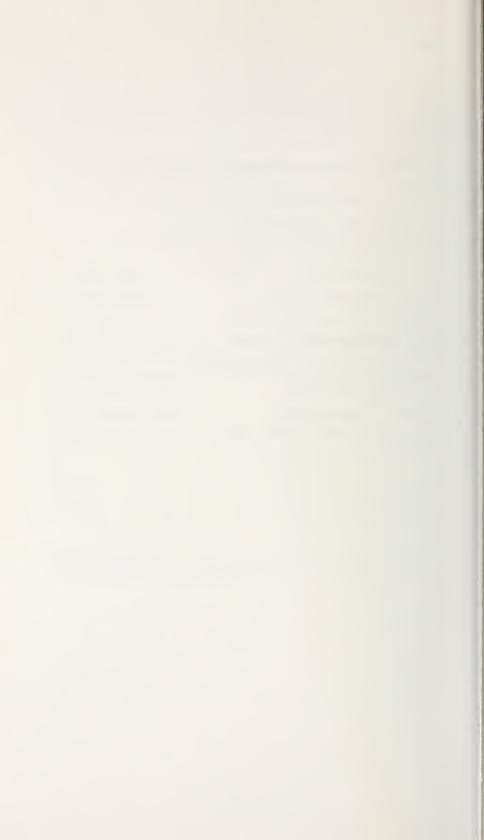
<sup>a</sup>not including wide range of high temperature corrosion resistant steels useful as reactor vessels, piping, etc.

Finally, in table 5 are shown those SRM's now in process which will benefit users in the field of nuclear science and technology.

Table 5. SRM's in process of interest to nuclear field.

- 1. Uranium isotopic series
- 2. Boric acid neutron absorber
- 3. Neutron flux beads
- 4. Neutron flux wires
- 5. Uranium metal assay
- 6. Boron glass neutron absorber
- 7. Plutonium sulfate intermediate purity assay
- 8. Plutonium metal isotopic composition
- 9. Radioactive isotopes

Point source series; I-129; Kr-85; Th-228 - others



# PROGRAMS AND SERVICES OF THE RADIOACTIVITY STANDARDS GROUP AT THE NATIONAL BUREAU OF STANDARDS

S. B. Garfinkel

The radioactivity standards group at NBS has the mission of developing, preparing, and maintaining radioactivity standards. These are standard reference materials, as contrasted to written standards. In addition to the distribution of these standards, we also provide a calibration service for almost all of the radionuclides which we have worked with, and in particular, for those radionuclides which we have standardized in the past, but no longer distribute. This service is available on a cost basis to individuals, private companies, government agencies, research laboratories, etc. Some of the standard reference materials have been dropped from distribution either due to lack of demand, or because they are available from commercial distributors who have demonstrated their willingness and competence to maintain their calibration facilities in a good enough order to meet the requirements and demands of the public.

The following table illustrates those standards which are currently available, and/or are being developed as new standards, and which should be available by the end of 1968.

The alpha-particle sources, polonium-210, americium-241, and plutonium-238 are practically weightless deposits on 1-inch-diameter monel disks. The electron capturers, betaray emitters, and some of the gamma-ray emitters are available in solution form, generally in 5-milliliter flame-sealed glass ampoules. Inasmuch as the gamma-ray sources can be used without opening the ampoule, these standards are certified both as to total activity, and also in terms of activity per gram of solution, should the user wish to open the ampoule to make weaker sources. This is, however, an uneconomic practice for the user. He would do better to place some stock solution, of similar radioactive concentration of 123

	RADIUM SOLUTIONS:	For Radon Analysis: 10-8 gram _0	10 <sup>-7</sup> gram 10 <sup>-11</sup> gram Blank	For Y-ray Measurements: 10 intensities from 0.1 to 100 µg	UNDER DEVELOPMENT:	Nickel-63 8-ray solution standard	Krypton-85 Y-ray gas standard Plutonium-238 ¤-source	DISCONTINUED STANDARDS: Scandium-46	Strontium-89 Strontium-89
Radioactivity Standards	B GAS STANDARD:	Krypton-85 Y-RAY POINT SOURCES (Energy in MeV):	<pre>Sodium-22 (0.511, 1.274) Manganese-54 (0.835) Cobalt-60 (1.173, 1.332)</pre>	<pre>Zinc-65 (1.114) *Yttrium-88 (1.836) Niobium-94 (0.702, 0.871)</pre>	*Niobium-95 (0.765)	Cadmium-109 (0.088) Cesium-137 (0.662)	Cerium-139 (0.166) *Mercury-203 (0.279) Thorium-228-Thallium-208 (2.615)	Contemporary Standard	for Carbon-14 Dating Laboratories: Oxalic Acid
	a SOURCES:	Polonium-210 Americium-241	<pre>8, Y, EC SOLUTION SOURCES: Hydrogen-3 (water, 2 intensities; toluene)</pre>	Carbon-14 (water, benzoic acid in toluene hexadecane)	Sodium-22	Chlorine-36 Iron-55	*Niobium-95 Tin-113-Indium-113m *Iodine-125 *Cerium-141	Cerium-Praseodymium-144 Promethium-147	*Mercury-197 *Mercury-203

Radioactivity Standards

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## Government-NBS Radioactivity

\*Not always available, due to combination of half-life and customer demand.

#### Garfinkel

the same radionuclide, in a similar ampoule and compare it to the standard, with any kind of detector, and then prepare his sources from the now calibrated stock solution. The electron-capturer and beta-ray standard solutions, on the other hand, are calibrated only in terms of activity per gram of solution, as it is almost always necessary to open these ampoules in order to use these radionuclides.

The gamma-ray point-sources consist of aluminum annuli 5.5-centimeter outer diameter, 3.9-centimeter inner diameter, over which are stretched a layer of adhesive polyester tape, about 0.008-centimeter thick. The radioactive material is deposited in solution form onto the center of this mount, and after drying, is covered by another layer of the same kind of tape. These standards are certified in terms of the number of gamma rays of a given energy, emitted per unit time. The range covered by this series extends from 88 keV to 2.62 MeV.

Almost all of the gamma-ray emitters are calibrated by  $\beta-\gamma$ ,  $X-\gamma$ , or  $\gamma-\gamma$  coincidence counting techniques. In those cases such as cesium-137-barium-137m, tin-113-indium-113m, and cadmium-109-silver-109m, where the long isomeric states preclude coincidence-counting techniques, it is necessary to use calibrated detectors which have been standardized by other calibrated radionuclide sources.

During the past several years, we have been able to do a better job of impurity analysis, as a result of the availability of the high-resolution lithium-drifted germanium detectors. We have thus been able to detect (and remove) small amounts of contaminants, which if long-lived, could "devalue" the standard in more ways than one!

There is one type of impurity, that is, an isotopic one of long half-life, that we could not consider removing until the very recent past. Consider the case of 20,000 year niobium-94, which is prepared by neutron irradiation of the 100% abundant niobium-93. In this process, there is also produced a considerable amount of 30 keV, 10 year niobium-93m. With the newly installed NBS isotope separator, we hope to be able

to produce carrier-free niobium-94 from which pure standards will be made. The present niobium-94 standards contain niobium-93m, and the content is so noted on the certificates. When the pure material becomes available, we will then be able to use  $4\pi\beta-\gamma$  coincidence counting as an independent check on the  $\gamma-\gamma$  coincidence counting which we used on the current batch of niobium-94 standards.

We have also been paying more attention to the statement of errors on our certificates. Specifically, we are giving values for both the standard (statistical) error at a given confidence level (usually 99%), and also for an estimated maximum uncertainty due to systematic errors.

From time-to-time, we get complaints from persons who have purchased a standard from us and who can not reconcile their measurement results with ours. In almost all cases, there has been a misunderstanding on the part of the purchaser of either the decay scheme or the half-life (we are now including a recommended value of  $T^{\frac{1}{2}}$  on all new certificates), and we have been able to resolve these difficulties quite easily. However, we also get complaints of customers of some commercial firms who claim "NBS traceability" of their standards, but whose products leave something to be desired. NBS is neither like the Federal Trade Commission nor the Food and Drug Administration. We have no "police powers", and there is not much we can do vis-a-vis these firms. What we can do is to suggest to their customers that they ask these firms how they maintain their calibrations in order, how often they do so, and so on. I understand that in a subsequent talk, some data will be shown that indicate "standards" purchased from one firm, actually had deviations of up to 50% of their stated values, when they were supposed to  $\pm 5\%$  sources.

# PRESENT AND FUTURE NEEDS OF THE AEC FOR STANDARDS FOR REACTOR DEVELOPMENT AND TECHNOLOGY J. W. Crawford

I want to thank the officers and members of the American Chemical Society for the opportunity to discuss the Commission's needs for standards for reactor development and technology. It is encouraging to see the keen interest in nuclear standards evidenced by your having organized a Symposium on Standards for Nuclear Chemistry and Technology, and especially one of such comprehensive scope. As you may know, AEC Commissioner James T. Ramey, Milton Shaw, Director of the Commission's Division of Reactor Development and Technology, and others in the Commission have been forcefully emphasizing to the nuclear industry - in all its segments - the need for development and application of proven engineering standards and other quality assurance practices. It is a pleasure, therefore, to address you, who appreciate the value of nuclear standards and who are in a position to exert a strong influence upon the quality of the materials, the processes, and the components used in our reactor development programs.

My remarks will be concerned with the programs that we in the Division of Reactor Development and Technology are pursuing. I shall point out the areas in which we are strengthening our standards and quality assurance practices to better assure the success of these programs and to better assure the availability for safe and reliable operation of the test and demonstration facilities associated with them.

The need for strengthening action of this kind has resulted from a number of interrelated circumstances which have underscored the importance of technical standards. A rash of problems, failures and delays had been encountered in the Commission's reactor development programs; and, I may add, in industry reactor programs also. These difficulties were proving costly, not only in dollars, but in terms of program delays as well. Such problems have even contributed to can-127 cellation of some Commission programs with consequent loss of expected technology advances for which these programs were undertaken.

The cause of the problems referred to was in large degree the lack of sufficient engineering attention. Many involved essentially conventional or non-developmental materials, processes, components, and systems. Most could have been prevented by application of proven engineering methods and practices.

Engineering standards provide the means by which the customer can define technical requirements to reactor plant suppliers, component vendors, architect-engineers, and plant constructors, and then exact compliance with these requirements. Engineering standards are also an important means of defining the existing base of technology upon which to build advanced reactor development programs.

We in the Commission are taking positive steps to use and develop standards for such programs. Where recognized codes and standards for design, fabrication, construction, operation and maintenance exist, we are insisting that they be used. Where existing standards are inadequate, we are supplementing or modifying them. Where standards are lacking, we are causing them to be developed.

Unfortunately, many available standards are incomplete in their scope of coverage and inadequate in stringency of requirements for application to reactor programs. New and upgraded standards are needed to establish more exacting requirements throughout all segments of reactor development programs. Standards are needed for nuclear and structural materials; for major reactor components, such as pumps, valves, heat exchangers; for fuel elements, and for instruments and controls. Standards are needed for fabricating, processing, installing and protecting materials and components. Standards are needed for long term operation and maintenance of reactor test facilities and nuclear power plants. Quality assurance program standards are needed to

#### Crawford

assure design adequacy and conformance to engineering requirements. Detailed technical standards are needed for cleaning, welding, calibrating, nondestructive testing and acceptance testing.

Now it is appropriate to describe what the Division of Reactor Development and Technology (RDT) has been doing to help meet these needs. A broad RDT standards program has been established and given the high priority and the means for developing acceptable engineering standards and quality assurance practices for application to the Commission's reactor development programs. This program is being carried forward to varying degrees on all RDT programs by our national laboratories and contractors. In addition, we are participating in the work of the many nuclear standards committees and organizations including the United States of America Standards Institute, the ASME, the ASTM and others. We are coordinating our activities with the Commision's regulatory organization. Key areas of cooperative efforts include pressure vessels, piping, pumps and valves, quality assurance system requirements, in-service inspection, and reactor protection systems.

Lead responsibilities for the preparation of RDT standards have been assigned to the Oak Ridge National Laboratory (ORNL) and the AEC's Liquid Metals Engineering Center (LMEC) at Santa Susana, California. The Oak Ridge standards effort is drawing together and consolidating current proven technology, engineering standards, and quality assurance practices, and will develop, validate and maintain current, basic uniform RDT standards for water-cooled reactors. Such RDT standards are intended for direct application to priority AEC water reactor projects such as the Loss-of-Fluid-Test (LOFT) and Power Burst Facility (PBF) projects. These two projects, as you may know, are important elements in our reactor safety research program.

Earlier it was indicated that the existing base of technology for water-cooled and other types of reactors pro-

### AEC-Reactor Development

vides the foundation upon which to build advanced reactor programs. To bring this experience together for the Commission's high priority Liquid Metal Fast Breeder Reactor (LMFBR) program, LMEC is drawing together and consolidating the different or modified basic standards applicable to the LMFBR program. Such RDT standards are intended for direct application to AEC priority LMFBR programs and projects such as the Experimental Breeder Reactor II (EBR-II), Sodium Pump Test Facility (SPTF), and the Fast Flux Test Facility (FFTF).

These standards efforts embrace areas of design, materials, equipment, processes, fabrication, construction, quality assurance, testing, maintenance, repair, and operation of reactor systems. In the materials areas, for example, material standards being prepared are of two general types: (1) those prepared to supplement the requirements of existing material standards, e.g., ASTM; and (2) new standards based on current technology. These RDT standards encompass alloys based on iron, nickel, aluminum and zirconium in a wide variety of cast and wrought forms. The documents are patterned after existing standards, such as those of ASTM. Some of these material standards provide options which extend their applicability to both water-cooled and liquid-metal-cooled reactor systems. Of primary interest to the LMFBR program is the development of those material standards which are applicable to pressurized components, such as valves, flanges and fittings, which must contain liquid metal at high temperatures.

As one might expect, the fields of chemistry and chemical engineering are intimately and widely associated with the RDT standards for reactor components and facilities. Standares are being developed, for example, for establishing and determining the chemical composition of a variety of materials for nuclear service. Methods of surveillance are being developed for the investigation of corrosion, erosion, radiation-induced distortion and irradiation embrittlement of materials such as those used on pressure vessels. Cleaning

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and cleanliness requirements are covered in detail and as a part of practically every reactor component standard. Contamination control is covered in several standards dealing with subjects such as activated charcoal filters and decontamination practices.

Insofar as the LMFBR program is concerned, one of the most important efforts is directed toward the development of standards related to sodium technology for application to the components and systems in the fast reactor environment. For example, sodium quality requirements in terms of significant impurities, tolerance limits and analytical methods are being established. Standard procedures are being developed to verify that sodium purity is attained and maintained in operating nuclear systems.

Recently, significant strengthening steps have been taken in the nuclear industry toward the development of material, equipment, process and plant standards for reactor applications. However, when one compares the relatively small number of acceptable standards that are presently available with the very large number that are needed for a typical reactor power plant, it is evident that considerable work remains to be done. The task is one that requires strong efforts by the nuclear industry through the standards programs of professional societies and trade associations. The results of such efforts should prove useful to the Commission's regulatory organization which has the responsibility for establishing and enforcing requirements to protect public safety.

For all these standards efforts to succeed, strong management attention is needed both in their development and in their application. The urgent need for upgraded standards must be understood, apathy toward their application overcome, and a systematic disciplined engineering approach adopted. "Business as usual" and "that's the way we've always done it" attitudes must give way to the practices which have been proven effective in successfully carrying out large and complex nuclear power plant projects in the past.

## AEC-Reactor Development

In the Commission's reactor development and technology programs we are insisting on this kind of strong management attention and a systematic ordered engineering approach. Such emphasis, including requirements for developing, adopting and enforcing engineering standards and practices, has resulted in delays and increased costs to projects already underway. However, experience in reactor development programs demonstrates conclusively that application of a systematic approach will better insure success. It will result in more realistic and predictable cost and time estimates of associated reactor plant and test facility projects. It will correspondingly reduce the likelihood and magnitude of unplanned and recurring engineering problems, cost over-runs, and delays, or even substantial loss of the investments made. Actions that place adequate emphasis on effective engineering standards and quality assurance practices are and will continue to be essential to assure safety, maximum benefit from an extremely large national investment, and technical and economic success of the Commission's reactor development programs.

# STANDARDS FOR NUCLEAR SAFEGUARDS NATIONAL AND INTERNATIONAL Samuel C. T. McDowell, Ralph J. Jones LeRoy R. Norderhaug ABSTRACT

One of the major factors in an effective nuclear safeguards system is the accurate quantitative knowledge of the special nuclear material in the nuclear fuel cycle. Laboratories and analysts can refine techniques and attain high precision but without an appropriate standard reference material there can be no measure of the deviation from the "correct" or "true" value. The AEC has long recognized the need for such standard reference materials and since 1957 has conducted a joint program with the National Bureau of Standards (NBS) to develop and distribute the standard reference materials needed for accurate measurements of uranium and plutonium in the nuclear fuel cycle. This paper discusses that program, the standards that have been made available and international acceptance of these standards. The paper also will consider the future of the nuclear industry, for example, the advent of high flux breeder reactors, and needs for standard reference materials dictated by such developments.

## INTRODUCTION

An effective safeguards program is dependent on three elements of material control:

Physical security such as locks, seals, and guards, Surveillance or monitoring by either inspectors or automatic instrumentation, and

Accountability which involves measurements of material and audits of records.

Just as a chain is only as strong as its weakest link, so no safeguards system can be more precise than the material standards on which that system is based. The United States Atomic Energy Commission has long recognized that a sound 133

### AEC-Safeguards

standard reference material program is essential to proper control of nuclear material. In September of 1948 the Commission formed the Fissionable Standards Committee. This Committee was asked to recommend and coordinate preparation of fissionable material standards for use in the Commission's laboratories.

The Committee recommended that pure uranium-235 and uranium-238 be obtained for use as "primary generative materials". These materials were prepared by electromagnetic separation at Oak Ridge and were then used to develop a series of internal standards with various isotopic enrichments.

The primary generative materials and the series of uranium isotopic standards represented the AEC's first attempt to provide the analytical bench marks so necessary for accurate chemical and isotopic analysis.

# ADVISORY COMMITTEE

As the number of private nuclear companies conducting research increased, the AEC expanded the scope of its standards program. In 1957 the Commission established the Advisory Committee for Standard Reference Material and Methods of Measurement. This Committee reflected the many varied disciplines of a growing nuclear industry. Its members represented academic and industrial interests as well as other government agencies.

Until 1967, the Advisory Committee provided liaison between the AEC and private industry. The Committee brought to the AEC a unique awareness of the measurement problems and reference material requirements confronting the private nuclear industry.

In 1967 the Advisory Committee was replaced by the broader based Advisory Committee for Nuclear Materials Safeguards.

The Advisory Committee for Standard Reference Materials and Methods of Measurement was charged with a two fold task:

- Prepare a comprehensive handbook of analytical methods for measuring nuclear material.
- Recommend additional standards necessary to assure accurate analysis of nuclear material.

To implement the recommendations of the Committee, the AEC enlisted the aid of the National Bureau of Standards. The Bureau's unique experience with chemical standards as well as an impeccable reputation for exactness made that organization most desirable for certification and distribution of nuclear material standards. Funded by the AEC until 1963, this certification program is now supported solely by NBS.

SELECTED MEASUREMENT METHODS

In 1963 the handbook "Selected Measurement Methods for Plutonium and Uranium in the Nuclear Fuel Cycle" was published. This book, edited by Ralph J. Jones of the Atomic Energy Commission, incorporated the collective experience of the Committee as well as of a great many other consultants. "Selected Measurement Methods" was issued as a Technical Information Division Report No. 7029. It has proven to be a valuable reference tool and can be found in analytical laboratories throughout the world.

At the Committee's recommendation, the AEC's New Brunswick Laboratory evaluated the measurement methods listed in the above handbook. A New Brunswick Laboratory publication, NBL-231, reports the practical precision and possible biases associated with these methods.

ADVISORY COMMITTEE RECOMMENDATIONS

Uranium oxide, U<sub>3</sub>0<sub>8</sub> was the first chemical standard for uranium distributed by the National Bureau of Standards. At the Advisory Committee's recommendation, AEC laboratories undertook a program to determine the stoichiometry of this compound. The stoichiometry was found to be unpredictable except when prepared under carefully controlled conditions. Although suitable for a working standard, this oxide did not meet the NBS requirements for a primary standard. Today uranium oxide is prepared by the New Brunswick Laboratory and is distributed as a working standard for uranium.

The Committee recommended that pure uranium metal be distributed as a primary chemical standard in lieu of the oxide. At present, the AEC has on hand an 80 kg block of high purity uranium metal. This block must be subdivided into units suitable for distribution as a primary standard. A pilot run on a similar block of uranium metal has been conducted at the Paducah Gaseous Diffusion Plant. A report of the resulting impurity levels is being prepared.

At present the National Bureau of Standards also distributes ultra-pure plutonium metal as a chemical standard. This expensive metal, prepared for the AEC by Los Alamos Scientific Laboratories, contains less than 100 ppm "impurities".

To reduce the overall cost of plutonium standards, the Advisory Committee recommended that a working standard be developed for plutonium as well as uranium. Three compounds were analyzed under various aging and laboratory conditions. Samples of the three compounds,  $Pu(SO_4)_3$ .  $4H_2O$ , anhydrous  $Pu(SO_4)_3$  and  $Cs_2PuCl_6$ , were sent to a number of laboratories for analysis. As a result of this round-robin analysis the Bureau will soon be distributing plutonium sulfate tetrahydrate as a plutonium working standard.

In addition to the primary and working chemical standards a series of 16 uranium isotopic standards is available from NBS. These standards range in enrichment from 0.5 to 93% uranium-235. The National Bureau of Standards also offers a plutonium isotopic standard containing approximately 90% plutonium-239 and 8% plutonium-240. The Atomic Energy Commission and the National Bureau of Standards are now considering the need for two additional plutonium standards. These, with plutonium 240 concentrations of 12 and 20% will be characteristic of the plutonium being recovered from the high burnup fuel of pressurized water and boiling water re-

# McDowell, Jones, Norderhaug

actors. In the future, as plutonium recycle and breeder reactors come on line, the AEC will assess the need for plutonium standards having still other isotopic ratios.

# INTERNATIONAL ACCEPTANCE

At the 1965 Euratom meeting to discuss high precision mass spectroscopy,[1] the participants generally agreed to use the NBS series of uranium isotopic standards for calibration of their instruments. Of the total nuclear material standards currently distributed by the National Bureau of Standards, 24% are to foreign recipients.

### SPECIAL PURPOSE STANDARDS

In addition to those standards planned for distribution by the Bureau, the AEC recognizes the need for others. Special purpose calibration standards will be needed in conjunction with the Commission's safeguards research and development activities.

Research projects are currently underway to develop non-destructive assay techniques for uranium and plutonium. Pulsed neutron and gamma interrogation techniques will require a variety of new standards. Standard mockups of fuel elements and barrels containing uranium and plutonium scrap will be needed to calibrate future instruments. High resolution gamma spectroscopy promises to be a useful tool for identifying fuel material and for verifying the reported irradiation history of spent fuel. To analyze complicated spectra, gamma spectroscopists need pure samples of individual isotopes of plutonium, uranium and selected fission products.

The latest designs for chemical reprocessing plants emphasize instrumented processing. As new plants are built new special purpose standards will be needed for calibrating automatic control devices.

 [1] EANDC - 53"S", "Round Table on High Precision Mass Spectrometry and Alpha Counting", November 29 - December 2, 1965.

# AEC-Safeguards

## SUMMARY

Each year the National Bureau of Standards distributes over a thousand standard reference samples of nuclear material. As new fuel forms and analytical techniques are developed, new isotopic standards will supplement the present series. Uranium metal as well as plutonium metal will soon be distributed as a primary chemical standard. Economical working standards will afford chemists an added degree of flexibility in their analytical procedures.

The success of the AEC's Standard Reference Material Program depends on the continued support of the National Bureau of Standards and of analytical laboratories such as New Brunswick Laboratory. In the final analysis, the Commission's safeguards system is dependent on the availability of a wide range of standards. To be effective, a safeguards system requires chemical and isotopic reference materials of the very highest precision and accuracy.

# STANDARDS IN RADIOISOTOPE DEVELOPMENT William E. Mott and Warren K. Eister

In the Division of Isotopes Development, we are acutely aware of the needs for standards as defined by the organizers of this Symposium. In many respects the progress we make is strongly dependent on the availability of standards: standard data, standard materials, standard specifications, and standard procedures. Without standards it is as difficult to imagine an effective research and development program on radioisotopes and radioisotope applications as it is to imagine the effective utilization of the resulting technology. The objective of this paper is to call attention to the needs for standards in the development and utilization of radiation and radioisotope applications.

### RADIOISOTOPE PRODUCTION AND MATERIALS

Our radioisotope production and materials program would be fraught with difficulties were it not for existing standards. On the other hand, our lives would no doubt be much easier at times if we had more and better standards. A case in point is that of the production of sealed sources for process radiation applications. Design and manufacturing standards have helped to reduce product (and hence irradiator) costs while increasing product safety and flexibility of product use. But even so, there can be problems. To exemplify, trace amounts of moisture in sealed sources have caused swelling and rupture in some cases. Standard manufacturing procedures have been revised and new procedures added to eliminate these problems.

Perhaps one of the most obvious examples of an activity requiring standards is the production and processing of radioisotopes for medical diagnosis. The questions of quantity, purity, and chemical form are of vital interest so that control over all the production and processing factors back to the introduction of the basic raw materials is mandatory. In 139 short, the problem is intractable in the absence of a carefully thought-out standardization program.

In our radioisotope production program, activities of by no-means minor importance relates to the preparation of pure radionuclides for evaluations including the determination of half-lives and decay schemes, in short, the accumulation of standard data. The continuing work is largely concerned with radionuclides not yet in general use (e.g., iodine-123, gallium-68, iridium-ll3m), although, as new radiation detection systems become available, it is always instructive to relook at the old favorites in order to obtain more precise data. In the preparation of pure nuclides for these studies there is a very definite requirement for standard procedures. Standard data emerges when materials prepared by standard procedures are dealt with in a standard fashion.

ANALYSIS, EVALUATION AND CONTROL

Broadly speaking, the most important applications of radioisotopes today probably fall under the analysis, evaluation and control heading. Activities include radiography, tracing, gauging, activation, Mössbauer, and radioisotope Xray. To be sure, the timely adoption of standards and standardization methods and procedures, in this area has always had a lasting impact.

To give an understanding of the part that standards play in radiation analysis, evaluation and control applications, we have selected a relatively new analytical technique that is not in widespread, routine use; namely, radioisotope X-ray spectrometry. Although the needs for standards and standardization here are only just arising and are certainly not well defined, there is, nevertheless, much room for discussion.

As a starter, there is the question of <u>terminology</u>. We all know that it is seldom too soon at the beginning of a new endeavor to standardize on terms. Those in this field must develop a common understanding of such terms as "radioisotope X-ray spectrometry" and "central source geometry."

As with any analytical tool, good <u>calibration standards</u> can often be the key to success. Physical and chemical stability, homogeneity, and chemical composition are of course important. Also, the overall composition must be as similar as possible to the materials being analyzed in order to reduce errors due to matrix effects. Carefully analyzed samples of the material to be tested are best, but sufficient ranges of concentration of each element in question are often impossible to obtain especially for ore and mineral analysis. In alloy analysis, less than a dozen suitable steel samples are available from the combined sources of the National Bureau of Standards and its British counterpart, the Bureau of Analyzed Samples. Few samples of other alloys are available. A great need may be rapidly building in this area.

X-ray filters, an important part of nondispersive X-ray spectrometry, must be uniform, of precise mass per unit area, physically and chemically stable, strong, and of controlled chemical composition. Many methods of making filters exist. The best filters will probably evolve by natural selection. Filters currently used include: metal foils; powdered elements of chemicals encapsulated in epoxy, polyethylene, or polystyrene; metals electroplated on beryllium foil; and films deposited on Mylar or polypropylene foil.

Along with the calibration standards, and the filters when required, comes the need for <u>standard operating proce-</u> <u>dures</u> to be followed both during standardization and calibration and during routine laboratory and field use. The best instruments and standards yield little of value if incorrectly employed.

And what has been said about X-ray spectrometry applies equally well to such more-developed applications as nuclear moisture and density gauging. There are currently over 500 neutron moisture and gamma-ray density gauges being used by state highway departments in highway construction in the USA. These are in about 25 different models and come from six instrument companies. Some of these gages employ transmission, some backscatter techniques. And even when the same techni-

## AEC-Radioisotope Development

que is used, sources detectors, and source-detector geometries are likely to differ. The purpose of all is to give information from which soil compaction - an important factor in constructing highways that will stand up under specified loads - can be determined. As we learned long ago, in nuclear well logging, where the principles and problems are very similar, the ability to produce interpretable results, as well as results that can be intercompared, is a strong function of the materials and procedures adopted for instrument calibration and field operation.

### PROCESS RADIATION

In process radiation applications (food, medical supplies, chemical and physical systems) it is extremely important to know how much radiation is being absorbed per unit time in a unit volume of the material being irradiated. At present there are literally dozens of dosimetry systems in use and under investigation, and unquestionably there is a need for standardization. Standardization is complicated, however, because many factors enter into the selection of a dosimeter for a given application. Some of these are:

- a. dosimeter size in relation to target size;
- b. dosimeter range, energy response and linearity;
- c. dosimeter compatibility with the environment in which it is used, such as the ability to withstand high corrosive or erosive environments and extremes in temperature and humidity;
- d. effect of target size and thickness on distribution and mean energy of radiation;
- e. the form of the target material, i.e., whether liquid, gas, or solid;
- f. the purpose of the dosimeter, e.g., the dosimeter for initial source calibration requiring a high degree of accuracy would probably not be pertinent for process control;
- g. the type of radiation, i.e., whether alpha, beta, electron, gamma, or neutron; and

h. the source-target configuration.

All of this means that it is unrealistic to speak about a universal dosimeter in process radiation applications. The need is more for an expanded series of standard systems that can be applied in well-defined cases.

Another process radiation need is for source, facility, and operational standards that will assure safe and reliable operation of the facility. Generally, federal and state requirements such as in Parts 10, 20 and 30 of the Code of Federal Regulations set the standards to be met. They do not always assure, however, that the material being irradiated remains stable under all radiation-imposed conditions. As an example, consider the radiation polymerization of methylmethacrylate in a wood substrate - to produce a wood-polymer ma-The synergistic effect of heat and radiation during terial. polymerization can produce in some circumstances a high exotherm which, if not properly controlled, can cause an explo-This actually happened in impregnated sion in the material. wood samples irradiated at very high electron dose rates. Fortunately, the samples were quite small and no damage to the facility or equipment occurred. Thus, it is not only the obvious safety aspects that must be anticipated by standard operation procedures in radiation processing, as further evidenced by the accident which occurred at BNL a few weeks ago. In this case, following the impregnation of concrete with acrylonitrile, the excess monomer was drained off and stored in a 55-gallon drum in an outside building. Apparently some constituent in the concrete reacted with the catalyst inhibitor in the monomer breaking it down. The drum exploded causing damage but no personal injuries. But again, had this event occurred within the irradiator a serious safety problem could have arisen. Needless to say, it would have been prudent to periodically monitor the inhibitor content.

The point is, safety standards must be considered not only in light of radiation safety, but in the context of the entire facility operation. When there is a myriad of products being irradiated, each separate application in itself must undergo an in-depth evaluation of possible accident potential, and hopefully, careful chemical and radiation engineering practices will enable continued accident-free operation of radiation processing systems.

### THERMAL APPLICATIONS

Next to be touched upon is thermal applications. In such applications the energy of decay from the radioactive material being used is converted into heat either in the source material itself or in a surrounding medium. The thermal energy so-produced is then utilized directly for heating or is subsequently converted into mechanical or electrical energy. The Division of Isotopes Development has the responsibility for Commission programs in which the end objective is either direct heating or the production of mechanical energy.

Thinking particularly of the future as broader applications requiring a multiplicity of units and devices expand, I see a very great need for standards: for standard materials, standard specifications, and standard procedures. Heat source capsules must be fabricated, loaded with fuel, welded, tested, and qualified. The very nature of the problem will make standardization the order of the day and the beginning will certainly be with the radiation-emitting fuel because of the restraints it puts on the total system.

To illustrate, let us consider the fuel for one of the possible applications of the future - the artificial heart. As many of you may know, any totally implantable artificial heart regardless of how powered will have the following components: a blood pump, a power source, a means of transmitting power to the pump from the power source, and possibly some controls. In a radioisotopically-powered artificial heart, the power source would consist of a radioisotope heat source, a thermal energy storage unit, and a thermodynamic converter. The most likely heat source fuel is plutonium-238. But not any plutonium-238 will do if the radiation out-

put of a heat source is to be minimized. For one thing, there are low Z impurities present in as-produced plutonium which give rise to neutrons by  $(\alpha, n)$  reactions. And there is the isotopic impurity plutonium-236, the decay products of which emit some rather hard photons. Our objective is a standard plutonium and a standard plutonium fuel form (metal, oxide, or nitride). About all we know at the moment is that the standard plutonium of the future will most likely be electrorefined. During recent work at the Los Alamos Scientific Laboratory on the electrorefining of plutonium-238 fuel, we have in routine operations obtained material having the highest elemental purity of any ever produced, (better than 99.98%), with excellent reproducibility from batch to batch. In contrast, the bomb reduction process even with the best quality control seldom gives better than 99.8% material.

Following standardization on fuel, the next steps will include the selection of the standard fuel form (or forms), the fuel form shape, the containment material, the encapsulation procedure, and finally the qualification procedure.

What might not be immediately associated with the artificial heart program is a need for standard dosimetry practices. Much that happens could depend on the results of radiation measurements made by different people under different conditions with sources having different emission characteristics. Neutron and photon spectra and dose rates will be measured in air from bare and shielded sources and in and around animal phantoms, animals, human phantoms, and perhaps someday even humans. Two plutonium sources, one of 16 watts and one of 24 watts, have been implanted in dogs since October 1967 and April 1968, respectively. External spectra and neutron and photon doses are being measured. Sources simulating the radiation from these two sources, but without the accompanying heat, have been implanted in several dogs. Spectra and dose rates are being measured. In the next few years many simulated radiation as well as actual heat sources will be implanted in additional animal phantoms, in animals,

and in human phantoms. Spectra and dose rates will be measured. It is difficult for me to imagine the effective utilization of the results of these many experiments and measurements without standardization.

#### SAFETY

Safety is a general ingredient in all standard procedures involving radioisotopes; however, it is so vital to radioisotope production and utilization operations that it deserves highlighting.

Except in tracer-type applications, containment is, of course, a prime consideration. An important activity is the development of standard testing and qualification procedures for sealed sources along with the establishment of guides for the application and transportation of such sources in a manner to insure source integrity under all foreseeable circumstances. That there is a need for constant vigilance is occasionally brought home to us. During our initial shipment of cobalt-60 encapsulated sources for the Hawaiian irradiator, corrosion of the sources occurred in the shipping cask during shipment and may be attributed to a lack of adequate standards for large-scale shipments by sea.

With radiographic cameras there have been no serious containment problems, but there have been several operator exposures resulting from an apparent failure of source retraction mechanisms. A study by Underwriter Laboratories was inconclusive, and the AEC Regulatory Division is now assessing the problem with the cooperation of manufacturers and users. While there have been no significant problems with industrial gauges, they are in such widespread use that a study has been undertaken to evaluate experimentally their characteristics under fire and other accidents.

### CONCLUSIONS

Based on the foregoing, it is concluded that our present needs include:

 The development of more effective reporting of methods and procedures for processing, calibrating,

and using radioisotope standards. This may involve the cooperative efforts of the supplier and user of these standards, along with the instrument manufacturer.

- 2. Standard materials and procedures for radioisotopes analysis, evaluation and control applications.
- An expanded series of standard systems for measuring the energy absorbed by products in radiation processing.
- Standard practices for measuring neutron and photon doses in air and in tissue.
- 5. Standards for encapsulation materials and for welding methods to assure containment of sealed sources of radioisotopes. This is particularly important for thermal energy applications at temperatures above 1000°C.

The Division of Isotopes Development plans to continue to support the development of effective standards. These are essential to the productive use of radioisotopes in our national economy.



# STANDARDS NEEDS FOR THE DIVISION OF BIOLOGY AND MEDICINE PROGRAMS William F. Marlow

In order to have an idea of the need for standards in the programs of the Division of Biology and Medicine, U. S. Atomic Energy Commission, it is necessary to know something of the organization of the Division and of the research programs conducted and sponsored by it. Within the Division are the Medical Research Branch, Biology Branch, Environmental Sciences Branch, Fallout Studies Branch, Radiological Physics and Instrumentation Branch, Civil Effects Branch and Technical Analysis Branch, as well as the Administrative and Program Coordination Branches. The research programs of the technical branches are broad and varied. The goal of DBM's biological, medical, and environmental research program is to develop the scientific knowledge needed for the full comprehension of possible short- and long-term consequences of the interaction of radiation with biological systems, from molecules of biological interest, to ecological, meteorological and oceanographic systems, including the radiation biology of space explorations.

The Division's programs are grouped in the following categories: Molecular and cellular level studies; Radiation Genetics; Somatic effects-general; Toxicity of radioelements; Environmental radiation studies; Radiological physics; Health physics, Radiation instruments; Combating detrimental effects of radiation; Chemical toxicity; Nuclear energy civil effects; Atmospheric radioactivity and fallout; Cancer research; and Selected beneficial effects (of radioisotopes and radiation). Projects under these categories are carried out in the AEC's laboratories, and, under contract, in the AEC's national laboratories, universities, research institutes, hospitals, private laboratories and other government agencies' laboratories.

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Much of this research is basic in nature, while the remainder is more applied research directly oriented to AEC's prime mission. It is impossible because of time limitations, and unnecessary, to go into details of these programs here to see the application and need in them for standards in all five categories that are being considered at this symposium. I shall try to give a broad picture of these needs, with some outstanding specific examples of the needs and, also, some examples of what work is being done to meet specific needs.

As can be readily appreciated, work in many of the above categories requires the analysis of a wide variety of sample media for many radionuclides, both natural and manmade, and for stable trace elements. These media include airborne dust, atmospheric gases, precipitation, surface water, sea water, soils, rocks, sediments and biological materials. The biological samples may range from the simplest of plants and animals to and including man. In the great majority of cases, the concentrations of the radionuclides are extremely low, and the trace elements of interest may be in the parts per million or parts per billion range. In order to assure accurate and reproducible analyses, both accurate standards and reliable standard analytical procedures are necessary. In many cases, a special procedure is needed for a specific radionuclide in a particular medium.

The analysis of many of these environmental samples are carried out by or under contract to the AEC's Health and Safety Laboratory in New York City. HASL's Director, Dr. John H. Harley, will give a talk in the next session on "Standard Requirements for Environmental Analyses." Therefore, I will not go further into that program, other than to give an idea of the radionuclides for which standards are required in this field. Figure 1 lists the radionuclides for which HASL purchase's standards and for which it can <u>not</u> perform primary standardization.

Figure 1 HASL PRIMARY STANDARD PROGRAM A. Standards Purchased (HASL cannot perform primary standardization)

Nuclide	Source
Н-3	NBS
C-14	NBS
S-35	RCC, IAEA
Fe-55	IAEA
Pm-147	NBS, IAEA
Ra-226	NBS

Figure 2 shows the radionuclides for which HASL purchases standards on which they must make further check. Figure 2 HASL PRIMARY STANDARD PROGRAM

B. Standards Purchased and Checked at HASL

Nuclide	Source
Na-22	IAEA
Sc-46	IAEA
Mn-54	RCC, IAEA
Co-57	IAEA
Co-60	NBS, IAEA
Zn-65	NBS, IAEA
Y-88	RCC
Sr-90	NBS (old)
I <b>-</b> 125	NBS, RCC
Sb-125	RCC
Pb-210	Nuclear Chicago, RCC
Am-241	IAEA

Figure 3 gives the longest list - those radionuclides which HASL purchases, from which it makes its own standards.

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Figure 3 HASL PRIMARY STANDARD PROGRAM

C. Radionuclides Purchases Unstandardized and Standardized

at HASL			
Nuclide	Source	Nuclide	Source
Na-24	ORNL	Ce-144	NSEC
Ca-45	RCC	W-185	RCC
Cr-51	RCC	T1-204	ORNL
Fe-59	NSEC	Po-208	RCC
Sr-89	ORNL, RCC	Ra-228	RCC
Zr-95	ORNL	Th-228	RCC
Nb-95	RCC	Po-210	RCC
Ru-103	NSEC	Pu-238	ORNL
Ru-106	RCC	Pu-239	LASL
Cd-109	LASL	Transplutonium	ORNL
Cs-137	ORNL*	Short-lived	ORNL
Ce-141	NSEC		
	Nuclide Na-24 Ca-45 Cr-51 Fe-59 Sr-89 Zr-95 Nb-95 Ru-103 Ru-106 Cd-109 Cs-137	Nuclide         Source           Na-24         ORNL           Ca-45         RCC           Cr-51         RCC           Fe-59         NSEC           Sr-89         ORNL, RCC           Zr-95         ORNL           Nb-95         RCC           Ru-103         NSEC           Ru-106         RCC           Cd-109         LASL           Cs-137         ORNL*	Nuclide         Source         Nuclide           Na-24         ORNL         Ce-144           Ca-45         RCC         W-185           Cr-51         RCC         T1-204           Fe-59         NSEC         Po-208           Sr-89         ORNL, RCC         Ra-228           Zr-95         ORNL         Th-228           Nb-95         RCC         Po-210           Ru-103         NSEC         Pu-238           Ru-106         RCC         Pu-239           Cd-109         LASL         Transplutonium           Cs-137         ORNL*         Short-lived

\* IAEA Standard also compared.

Figure 4 shows the radionuclides HASL purchases for use as tracers in determining chemical yields in analytical procedures.

Figure 4 HASL PRIMARY STANDARD PROGRAM

D. Tracers

Nuclide	Source
Be-7	NSEC
Sr-85	NSEC
Ba-133	ORNL
Pu-236	UCRL

These tracers do not need to be standardized, but must be checked for purity before use. I realize that I am taking substance from Dr. Harley's field of discussion in giving these figures, for which I thank him, but they demonstrate very well the gamut of radionuclides involved throughout the DBM programs.

For the stable trace elements, many of which are determined by activation analysis, special standards, often invol-

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ving the medium in which the element is to be studied, are needed, as well as standard analytical procedures. Again, another speaker, Dr. Vincent Guinn, is scheduled to go into detail on this subject in a later session, so I will not pursue it further here.

Much work to develop standards and standard procedures is carried out by researchers in the course of their work involving the radionuclides and samples in question. A considerable amount of work, especially for the development of analytical procedures, is sponsored directly by DBM. A few outstanding cases are the work at HASL on both standards and analytical procedures; at the Health and Safety Division of National Reactor Test Station, Idaho; at Battelle-Pacific Northwest Laboratory, and at the National Bureau of Standards. Part of the work at HASL, and the work at NRTS, PNL and NBS are particularly directed toward rapid radiochemical separation schemes, especially as applicable to Activation Analysis samples. Under a contract to DBM, a group at the Massachusetts Institute of Technology is carrying out studies to make a systematic assessment of the application of activation analysis to forensic and clinical medicine. Work includes instrument development; testing and calibration; development of suitable procedures for tissue sampling and chemical separations; compilation of a reference library of standard spectra; establishment of a tissue bank; and analysis of a number of human tissues for a variety of elements. I would like to add an interesting note here. At PNL, during their study of a cellulose air filter medium, commercially known as IPC-1478, the investigators have found that this material has a uniform very low content of the elements antimony, cesium, cobalt, iron, scandium, silver, sodium and They therefore propose that the IPC-1478 be considered zinc. as a biological standard for activation analysis for the above elements. Since this work will be reported in detail by the investigators at The 1968 International Conference: Modern Trends in Activation Analysis at the National Bureau of Standards this October, I will not discuss it further.

# AEC-Biology and Medicine

Another DBM sponsored work that is of interest here was the compilation several years ago by an Ad Hoc Committee of procedures for the determination of very low concentrations of radionuclides in a variety of media. The object was to provide laboratories and regulatory groups with reliable procedures to determine that facilities handling radioactive materials were being operated within applicable safety standards and regulations. These procedures were tested for their accuracy and reproducibility in several cooperating laboratories. In case anyone is interested in this compilation, which does not have a formal document number, a few copies are still available at DBM.

In the field of whole body counting, standard reference samples are necessary, including absolute reference standards and "working standards," both the point-source type and the "phantom" type. The fulfillment of these needs has not always been easy. The following case points this up rather remarkably. In the Autumn of 1961, when heavy nuclear testing was suddenly resumed by the USSR, DBM instituted a program to measure the amounts and times of increases of cesium-137 in humans in different locations in the United States. About ten laboratories located in various geographical areas of the U. S. which had whole body counting facilities began counting groups of individuals on a weekly basis. We wanted to see if the arrival of waves of nuclear debris could be determined at the various locations across the country. After about nine months of this program, it became very obvious that only a few of the laboratories could do these measurements on a quantitative basis. Some could not quantify their data at all, and most experienced very erratic results. We therefore undertook, in collaboration with a group of experts, to set up a standardization program and obtain the necessary standards. For absolute measurements, the Radioactivity Section of NBS undertook to prepare for us sets of essentially mass-less, absorption-less standards of cesium-137 of the conventional one inch disc-and ring type, at count-

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ing levels appropriate to our needs. This proved much more difficult and time-consuming than anticipated, but the sources were produced. In addition, we desired essentially "pointsource" standards in lucite rods which could be handled routinely and repeatedly without adverse effects, and which had a fixed known geometry. The active source was to be no larger than one-eighth inch in diameter in a rod one-half inch in diameter. After consideration of the problem, NBS declined to try to produce them. The standards were then solicited from private laboratories. One laboratory spent many months and considerable funds on the project, but failed to come up with satisfactory sources. A second laboratory never got through the planning stage. Finally, a third laboratory, New England Nuclear, after truly many months of work and not a few failures, produced about twenty sets of a series of three sources, nominally 10,100 and 1000 nanocuries of cesium-137 each. These were used by the laboratories to calibrate their whole body counting systems, mainly for stability of performance, and response to a known absorptionless source of cesium-137. For actual quantification of levels measured in humans, "human sources" with known, or carefully-measured amounts of cesium-137, or cesium-134, were counted in different facilities. While some laboratories have developed phantom-type standards for their own use, I know of no individual one which is universally or even widely accepted throughout the community.

There is a very pressing need for standard radiation measuring instruments and standard procedures for their use, especially when used in peculiar environments. This need was very evident when DBM had to respond to the present urgent concern over the levels of air-borne radioactive contaminants in uranium mines. The instruments had to be designed to measure radon daughters, and then calibrated against radon daughters under levels and conditions existing in the mines. This was no easy task, but a three channel, portable gamma analyzer developed at the Massachusetts Institute of Technology is now being used quite successfully in these measurements.

# AEC-Biology and Medicine

In the field of instrumentation standardization, DBM's Standard Nuclear Instrument Modules program should be mentioned. With the advent of solid state electronics, the mechanical and electrical configurations of various instrument packages became numerous and incompatible. An AEC committee on Nuclear Instrument Modules was formed in 1964 and formulated designs and specifications to assure mechanical and electrical interchangeability. These specifications were first put out in AEC publication TID-20983 in July 1964, and have been updated in Revision 2, January 1968. These specifications have been widely and enthusiastically accepted. Copies of this publication may be obtained at the U. S. Government Printing Office.

The need for accurate standard reference data in the DBM programs is too obvious to require more attention here than it mention. Without such data, little could be done to calculate and determine the effects of ionizing radiation, particularly on biological tissues and systems, or calculate dose rates frc atmospheric and ground-level concentrations of radionuclides, both to exposed subjects and those in various types of shelter These data are also necessary for the dating of nuclear debris and the calculating of yields of nuclear devices from the concentrations of radionuclides in the atmosphere.

While this talk has only been a sketchy description of the biological, medical and environmental research program of the AEC's Division of Biology and Medicine, and the part that standards of various types play in that program, I hope that i has given you some overall insight into these matters, as well as an indication of what we are trying to do to solve our prob lems.

# RADIOACTIVITY SOURCE STANDARDS FOR PUBLIC HEALTH SERVICE NEEDS Harry E. Kolde and Patricia A. Cliggett ABSTRACT

U. S. Public Health Service uses and requirements of radioactivity standards, that is, materials with accurately defined radionuclide concentration are described. Major applications are studies of specific radionuclide contents in environmental media, experimental animals, and man, including radionuclides administered to humans for diagnostic purposes and physiological studies. The common primary use of standards is calibration of a variety of nuclear counting systems for efficiency determinations, spectra, and other performance measures. The user normally wishes to obtain these values with the highest possible accuracy. Standards are usually obtained from commercial suppliers. Examples of problems incurred with incorrect or inconsistent activity ratings, absence of certificate information, decay schemes, and other faults are described. Also noted is the unavailability of standardized forms of many radionuclides from domestic producers. Currently desirable types and forms of standard materials, such as gases and tagged media, are listed.

### INTRODUCTION

Within the Public Health Service, the two major groups concerned with radioactivity are the National Center for Radiological Health (NCRH) and the National Institutes of Health (NIH). The NCRH functions to evaluate long-term health effects of radiation and to prevent or control potential radiation hazards from nuclear device testing, nuclear reactors and facilities, industrial radioisotope users, and other man-made or natural sources of radiation.[1] These responsibilities require large scale sampling of all aspects of the environment, including man, for the measurement of radioactive contents.

### AEC-Public Health

At the NIH, as part of the rapidly growing field of nuclear medicine, radioactive substances are administered to many individuals for diagnosis of disease or study of physiological processes.[2] Prior to administration, the activity and purity of all radioactive preparations are checked at the NIH.[3] This assures that the proper dose of the proper radionuclide is given so that the subject's radiation exposure is minimized.

Many types of nuclear counting systems are employed in PHS laboratories to identify and measure an abundance of radionuclides. These include such instruments as gamma spectrometers with NaI(T1) or Ge(Li) detectors of many sizes and types, whole-body monitors, <u>in-vivo</u> organ or whole-body scanners, coincidence/anti-coincidence counters, and liquid scintillation counters.

A large variety of standardized radionuclides are used for calibrating these systems. Calibration standards are prepared to determine counting efficiency as a function of disintegration energy as well as sample or organ geometry, density, or depth in a body. In the case of whole-body monitors, calibration standards are administered to human volunteers or inserted in mannequins to relate counting efficiency to a body-size parameter such as subject weight or height. Standards of high radionuclide purity are necessary for spectral analyses. These spectra serve either as a reference for checking the purity of non-standardized forms of a radionuclide or as part of a library for resolving the components of complex mixtures of radionuclides.

DESIRABLE PROPERTIES OF STANDARDS

For most instrument calibrations, we prefer to work with absolutely standardized solutions because they provide a high degree of accuracy, purity, stability, and reliability. Their liquid form is convenient for standardization and preparation of simulated samples for calibrations. The radioactivity concentration of such standards is known absolutely since the disintegration rate is measured directly, i.e.,

without reference to any other standard of activity. Production methods are chosen to avoid or minimize radionuclidic impurities. Unavoidable impurities are identified and measured to determine their effect on the rating of the principal radionuclide. The chemical composition is chosen to assure homogeneity and minimum activity losses in preparation and storage. Each standard is processed carefully to maintain uniformity and purity. The random and systematic errors introduced by the measurement technique, impurities, and preparation are quantified so that the activity of the principal radionuclide can be stated within a given amount of certainty.

### PROBLEMS

We prefer to purchase our standards rather than perform absolute standardizations. Since 1962, the Radiation Safety Section of the NIH has obtained 180 standards of 32 radionuclides. During the same interval, the NCRH group in Cincinnati alone purchased over 150 standards of 55 radionuclides. Most standards were found to agree with advertised specifications, but problems in their use occur occasionally.[4]

The most common fault observed by us is poor agreement of calibration results from standards obtained from different suppliers. A recent intercomparison with  $^{57}$ Co standards may be of interest. This radionuclide with a 270-d half-life and photon energies of 122 and 136 keV is used as a calibration reference standard for measuring the important radiopharmaceutical nuclide, 6-h  $^{99m}$ Tc, which emits 140-keV photons.

During 1967, the NIH purchased 57Co standards from three U. S. suppliers. Two to six samples of each standard were analyzed with a counting precision of  $\pm 1$  to  $\pm 1.5$  percent at the 95 percent confidence level. A variation of 25 percent in the calibration results was observed (Table 1). Since the results exceeded the suppliers' quoted error, a sample of supplier C's material was sent to the National Bureau of Standards (NBS) for calibration. All results have been normalized to the NBS value. Conversation with supplier

Variability of Cobalt-57	Standards
Quoted	Discrepancy,*
Accuracy, %	%
<b>±</b> 5	3
<u>+</u> 6	11
<u>+</u> 6	-2
± 3	-14
on <u>+</u> 2	-
	Accuracy, % ± 5 ± 6 ± 6 ± 3

	Τa	able l	
Variability	of	Cobalt-57	Standards

\*Percent variation from result given by NBS calibration of supplier C standard.

B revealed that erratic malfunction of his instrumentation occurred in the time interval when his standard was prepared. This supplier shipped, at no charge, a replacement (B No. 2) which gave results within 2% of the NBS value.

A similar situation occurred when the NIHexamined a  $^{57}$ Co reference solution supplied by a radiopharmaceutical company. The company intended the recipient to use the solution for calibration of counting instruments for the assay of <sup>99m</sup>Tc eluates. The activity of an accurately measure sample of the preparation was compared to the NBS calibrated  $^{57}\mathrm{Co}$ standard. The NIH assay was 42 µCi/ml, compared to the supplier's labeled value of "55.78 µCi/ml". Use of the supplier's rating would have resulted in an administration of only 67 percent of the desired amount <sup>99m</sup>Tc to a subject. In some cases, the dosage would have been insufficient to provide the necessary diagnostic information.

The problem of selecting the best standard is also illustrated by the experience of the NCRH group in Cincinnati with  $^{140}Ba^{-140}La$ . During a two-year period, four absolutely standardized solutions were obtained from two suppliers. In this case, duplicate dilutions of each standard were prepared and duplicate samples of each were taken. When the difference between sample results was greater than the counting

precision of  $\pm 1$  percent at the 95 percent confidence level, additional samples were prepared. A variation of 33 percent was observed (Table 2). Shortly after receipt, the activity rating of solution No. la from supplier A was announced to be in error since the 15 percent greater activity of the  $^{140}$ La daughter when in transient equilibrium was not considered. The value for solution No. 1b represents the corrected result. A standard (No. 2) obtained later from supplier B showed 7 percent greater activity in comparison to standard No. 1b. Another standard (No. 3) was subsequently purchased from the second supplier and this revealed a gross difference in activity. Considerable re-evaluation by the supplier and NCRH did not resolve the discrepancy. The supplier issued a replacement (No. 4) which was adopted as the optimum standard after its activity rating was confirmed by  $4\pi\beta$  counting at Cincinnati.

	Variability of	Barium-140 Standa	ards
	Std.	Quoted	Discrepancy,*
Supplier	No.	Accuracy,%	%
А	la	± 1	-10
	lb	<u>+</u> 1	-3
В	2	+8, -4	+4
	3	+7, -2	-29
	4	+3, -2	0

Table 2 ariability of Barium-140 Standards

\*Percent variation from result given by std. No. 4 from supplier B.

One supplier was found to provide very inaccurate standards. A set of eight gamma-ray point sources was obtained by one NCRH laboratory to determine counting efficiency as a function of gamma energy. The sources appeared to yield unsatisfactory values and the Cincinnati NCRH group was requested to confirm the activity ratings. Our analysis was made by gamma spectroscopy, using calibration values obtained with absolute standards of the same radionuclides. Counting precision was maintained at less than  $\pm 1$  percent at the 95 percent confidence level. Table 3 shows the deviation of the supplier's ratings from the NCRH values. Only two, 109Cd and 137Cs, showed agreement. Three deviated by 11 to 15 percent and the other three were grossly erroneous. Although the supplier did not advertise the sources as standardized material, he did indicate that they were calibrated individually within an accuracy of  $\pm 5$  percent for an additional charge.

Radionuclide	Rating Deviation, %**
22 <sub>Na</sub>	+15
54 <sub>Mn</sub>	-64
<sup>60</sup> co	+13
65 <sub>Zn</sub>	+141
<sup>L09</sup> Cd	- 4
133 <sub>Ba</sub>	<b>-</b> 69
137 <sub>Cs</sub>	+ 4
144 <sub>Ce</sub>	+11

Table 3 Discrepancy of Set of Gamma-Ray Point Sources\*

\*Supplier accuracy quoted at ± 5%. \*\*% deviation = <u>supplier rating X 100</u> NCRH rating

Resolving discrepancies in these types of intercomparisons causes much lost time on our part. We also consider difficulties with activity ratings to be serious because the average nuclear medical or health department laboratory would ordinarily buy a standard from only one supplier, and inadvertent analytical errors could result. Most of these laboratories do not have the equipment, capability or time to perform absolute standardizations or sometimes even the means to verify the accuracy of purchased standards.

# Kolde, Cliggett

For state and local health departments, the problem has been partially corrected by the Analytical Quality Control Service (AQCS) of the NCRH. AQCS conducts large-scale methods cross-checks and intercomparison studies with he participation of many health departments. As an outgrowth of these efforts, dilutions of absolutely standardized solutions are provided upon request for instrument calibrations. This group purchases absolute standards from various suppliers and tests the original solution and its dilutions for accuracy of the radionuclide content and purity before issuance. The total radioactivity of each AQCS diluted standard is usually in the range of 10 to 500 nanocuries. Over 225 of these solutions were distributed during 1967 to 30 state and local health departments.

#### NEEDS

The lack of standardized forms of many radionuclides still exists. In 1967, Reynolds[5] reported that of 93 radionuclides commonly used, only 35 could be obtained as standards from domestic suppliers. A recent review of the catalogs indicates no improvement in the supply. Purchase of standards from domestic producers is preferred since delivery is usually faster and communications are easier should questions arise. Transportation and customs clearance delays makes it undesirable to import short-lived nuclides as 12.8hr. <sup>64</sup>Cu, 35-hr. <sup>82</sup>Br, or 6-hr. <sup>99m</sup>Tc.

Suppliers should also be alert to the need for standards of radionuclides beginning to be more frequently used. For example, applications of radiopharmaceuticals labeled with  $^{123}$ I,  $^{43}$ K,  $^{67}$ Cu,  $^{176}$ mLu,  $^{177}$ Lu, and  $^{169}$ Yb, were discussed at the June 1968 meeting of the Society of Nuclear Medicine.[6-10]

The only gamma-emitting gas available as a standard is  $^{85}$ Kr. Provision of a standardized 5.3-day  $^{133}$ Xe gas with its 81-keV photon would be desirable. Use of  $^{133}$ Xe in conjunction with 510-keV  $^{85}$ Kr could provide an efficiency <u>vs</u>. energy calibration curve for gamma analysis of gas samples obtained,

for example, in studies of nuclear reactor effluents. Xenon-133 has also become an important radionuclide in nuclear medicine.

In environmental studies, a need exists for standardized forms of environmentally-tagged media with low-level radioactivity contents such as  $^{90}$ Sr in bone ash or  $^{137}$ Cs in muscle ash. These can facilitate the calibration of counting systems for samples with densities other than water. In addition, they can permit verification of accuracy in methods development work and could serve quality control purposes in batch analyses of samples.

Regarding certificates, we note that at least three U. S. commercial suppliers have adopted to some extent the model format recommended by the NAS-NRC Subcommittee on the Use of Radioactivity Standards.[11] One of the three, however, only provides a general precision error term. Other suppliers still provide only such rudimentary information, as concentration, date, and lot number. Besides fulfilling regular needs, the data given in a comprehensive certificate often assists in diagnoses of apparent discrepancies. For example, because the supplier of the first barium-140 standard referred to in Table 2 indicated the assumption of the equal activities of the  $^{140}$ La daughter in equilibrium with  $^{140}$ Ba, the discrepancy was detected and brought to the supplier's attention.

Differences in decay scheme assumptions often explain inconsistencies between results given by standards from several producers. Agreement between suppliers on decay scheme data may be a solution. For example, the difficulties encountered with  $^{197}\mathrm{Hg}$  were partially solved by the suppliers meeting to select a decay scheme during the 1965 symposium on radioactive pharmaceuticals at Oak Ridge.

As an additional solution to some problems, we recommend the establishment of an independent technically-competent group that will undertake two prime tasks. First, determine the best available decay scheme data; communicate

these <u>readily</u> in an understandable form to all isotope producers, suppliers, and users; update the information as new facts come to light; and enlist the cooperation of all suppliers to adopt the recommended decay schemes. Secondly, this group should evaluate standards either submitted to it voluntarily by interested suppliers or by random market purchase. We believe that this would help to assure users that the materials advertised directly or indirectly as standards are accurately represented.

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### AEC-Public Health

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## STANDARDS FOR NUCLEAR INSTRUMENTS L. E. Packard

If you wouldn't mind a facetious approach to this presentation, I could make this really very brief by saying everything has been said here before. For example, we are another group working in the nuclear field who feel the need for standardization. We identify ourselves by some letters --A.N.I.M. We are organized and working. We are not very old, and yet we have had a fair personnel turnover. We all have full-time work other than standards. Our people who are doing the work deserve a lot of credit, but we wish we could get them to move more rapidly. And, finally, we are rather appalled at the amount of time and effort required to reach agreement on standards. I think we heard this whole story here yesterday.

But, seriously, I would like to go ahead and tell you in somewhat more detail our particular version of the story. First of all, I should define just who we are....the Association of Nuclear Instrument Manufacturers. Let me start by saying who we are <u>not</u>. We are not the Atomic Industrial Forum. We are not concerned with nuclear power per se. We are not concerned with radiation sources or accelerators per se. We are not producers of radiochemicals per se -- even though many of our members are also in this field.

The Association of Nuclear Instrument Manufacturers, Inc. is an independent and nonprofit organization. Its purpose is to represent the interests of its member companies which are engaged in the development and manufacture of scientific instruments for the measurement of nuclear radiation. A.N.I.M. presents a member-derived viewpoint to federal, state and local government agencies on issues affecting the growth, welfare and profitability of its member companies. In addition, it provides a vehicle for the development of industry-wide standards, guidelines for uniform quality, 167 warranties for service, improved marketing and exhibiting media, business statistics and industry public relations.

How do we define nuclear instrument industry? Let me just take a moment and read officially how we do that from our bylaws. The term "Nuclear Instruments Industry" includes but is not limited to any company engaged in developing and manufacturing and marketing commercial, i.e. catalog type, nuclear instruments in any of the following categories:

- 1. Multichannel analyzers
- Nuclear power supplies, scalers, amplifiers, count rate meters, single channel analyzers
- 3. Monitoring instruments
- 4. Detecting heads sold separately
- 5. Solid state detectors
- 6. Sample flow counting systems, manual and automatic
- Medical and biological counting systems, for in-vivo counting

These, incidentally, are essentially the categories that are used by McGraw-Hill in their industry survey of nuclear instruments.

Now I indicated we are not very old. We started this organization in 1965. It was based on an effort by McGraw-Hill to get the major companies in the field at that time together for a bookings project that they were undertaking as part of their overall reporting of the economy of the country. The initial companies that participated were those companies which had reported a sales volume in excess of \$1 million in this field in 1964. There were twelve companies. You might be interested just to hear who they were. This dates back to 1964 -- those companies which did more than \$1 million volume in this field: Baird-Atomic, Beckman, Eberline, Hamner, Nuclear-Chicago, Nuclear Data, Ortec, Packard, Picker-Nuclear, TMC, Tracerlab and Victoreen.

The formal incorporation of A.N.I.M. was set up in January of 1967. There are now twenty company members which among them represent approximately 75 to 80 percent of the

#### Packard

sales volume of commercial catalog-type nuclear radiation detecting and monitoring instruments. This is exclusive of industrial process control equipment, reactor instruments and radiation sources. And it is that percentage -- 75 to 80% -of those instruments manufactured in the U.S.A. The total amount is approaching \$100 million annually.

The following is the full current membership -- again as part of identifying who we are before we start to talk about what we do.

A.N.I.M. 1968 MEMBERSHIP

Baird-Atomic, Inc. Cambridge, Massachusetts Beckman Instrument Company La Jolla, California Canberra Industries, Inc. Middletown, Connecticut Santa Fe, New Mexico Eberline Instrument Corp. E. G. & G.\* Salem, Massachusetts Brooklyn, New York Eon Corporation Hamner/Harshaw Cleveland, Ohio Kaman Nuclear Colorado Springs, Colorado LeCroy Research Systems Corp. Elmsford, New York Nuclear Associates, Inc. Westbury, New York Nuclear-Chicago Corporation Des Plaines, Illinois Palatine, Illinois Nuclear Data, Incorporated Nuclear Diodes, Incorporated Prairie View, Illinois Nucleonic Corporation of America Brooklyn, New York ORTEC, Incorporated\* Oak Ridge, Tennessee Packard Instrument Company, Inc. Downers Grove, Illinois Picker Nuclear White Plains, New York Princeton Gamma Tech Princeton, New Jersey Technical Measurement Corp.\*\* North Haven, Connecticut Tennelec Instrument Company Oak Ridge, Tennessee Tracerlab Division of LFE Waltham, Massachusetts Victoreen Instrument Company Cleveland, Ohio \*E.G.&G. acquired Ortec in September 1967 \*\*Assets of company sold January 1968 AFFILIATE New York, New York McGraw-Hill, Inc.

### ANIM-Instruments

The following are the current officers and directors of the Association. The companies are represented by senior officers or division managers to ensure that firm positions can be taken at our meetings.

OFFICERS

	OFFICERS							
President	Frank H. Low	Picker Nuclear						
Vice President	Lyle E. Packard	Packard Instrument						
Treasurer	Rodman A. Sharp	Beckman Instruments						
Secretary	John M. Dempsey, Jr.	Baird-Atomic						
DIRECTORS								
Through 1968:	Richard J. Sandberg	Nuclear Data						
	*Rodman A. Sharp	Beckman Instruments						
	William J. Lepeska	Nuclear-Chicago						
	*Duane M. Mayhew	Victoreen Instrument						
Through 1969:	*Lyle E. Packard	Packard Instrument						
	Thomas L. Yount	E.G.&G./Ortec						
	*Frank H. Low	Picker Nuclear						
	Lee Dressner	Tennelec Instrument						
Through 1970:	John M. Dempsey, Jr.	Baird-Atomic						
	Donald G. Lowell	Eberline Instrument						
	*Fred W. Hannula	Tracerlab/LFE						
	Edward A. DeCrosta	Hamner/Harshaw						
	*Member of the Executive	Committee						

We have directors' meetings approximately quarterly and all of the work is done through committees. The committees that we have are STATISTICS, which give us the billings, bookings, profitability, etc., all on a concealed basis so no one knows the identity of these figures. They are all reported in, summarized, averaged in lump figures and fed back to us. The EXHIBITS committee which helps to set some standards both in the way our industry people conduct themselves at exhibits and the way participation is handled with the sponsoring organizations of exhibits, etc. The third committee is the STANDARDS committee which I'll go into in detail, of course. The fourth is LEGISLATIVE ACTION. The fifth is INTERNATIONAL TRADE PROMOTION which is a very important phase of our work.

#### Packard

We are contributing a substantial margin to the nation's favorable trade balance....something on the order of \$20 million a year. The last committee is MEMBERSHIP.

Now for the STANDARDS committee. Here I'd like to read from an annual report of the Association, actually paraphrase, I think would be a better way to put it, because this was originally written for general consumption and not for an audience of people concerned with standards and technical terminology of what a standard is. During the past two years, the STANDARDS committee has been developing design guidelines in several very important areas. These are being adopted as standards by A.N.I.M. They are to be used by the A.N.I.M. companies and are available to any others on request. In addition to the formal subcommittees, there were several ad hoc committees active. Reports on the activities of the subcommittees follow this introduction section. Here's one example of an item that we considered working on and dropped. The AEC had contacted us to determine our interest in film badge standardization. This is a little afield from nuclear instrumentation; but nevertheless the matter was considered and we decided to have nothing to do with that and I understand it is being handled by the National Sanitation Foundation at this point. Another thing that you might not think of as a standards activity -- we have developed a suggested form of standard instrument warranty. This document includes recognition of the various ancillary equipment that goes along with our instruments -- that seems to be a problem with the sale of commercial instruments -- and it also takes special consideration of items such as Geiger tubes, photomultiplier tubes, batteries, etc.

In another area closely related to standards, A.N.I.M. has been participating actively in a dialogue with several Governmental agencies and the offices of certain congressmen in the evolution of controls and/or standards for medical devices, which of course includes many nuclear instruments. Starting with the Williams Bill in August of 1965, A.N.I.M. has had representation at hearings in Washington to state its position and to report back to membership the status and situation. At present, there are two bills....and this was written in the spring of 1968. One is sponsored by the Administration and the FDA and is known as the Staggers Bill. The other is sponsored by Representative Reinecke of California. The former would impose Governmental control over medical instruments. The latter relies principally on the development through existing organizations of better standards to be generated and policed to a considerable extent by industry itself. A.N.I.M. has rather complete documentation on the medical devices legislation matter and it's all distributed to member companies.

Another small activity of the STANDARDS committee is to circulate standardized and appropriate patent clauses for use by the member companies in their personnel work with technical people of the companies.

The first of the STANDARDS subcommittees is on HEALTH PHYSICS INSTRUMENTS. Typically the subcommittees consist of a representative from each of the companies that manufacture the particular class of goods concerned. In this particular case Victoreen, Eberline, LFE/Tracerlab, and Texas Nuclear, part of Nuclear-Chicago, are represented. This subcommittee embraces one of the most active areas in nuclear standards work, since it concerns the physical protection and well being of those who work with radiation sources. It is imperative that all commercially available instruments be accurate and be interpreted identically by all users. Through A.N.I.M. efforts, advertisement and manufacturers' literature have begun to state energy response ranges for instruments measuring radiation dosage.

A.N.I.M. works in close cooperation with the AEC and in particular its Division of Biology and Medicine. It monitors efforts of individuals and groups in Government labs, who from time to time initiate action on portable instruments for health physics monitoring, and tries to extract the most meri-

torious aspects of these projects. A.N.I.M. also endeavors to keep in touch with and supply its members with information on United States and international standards.

The next subcommittee is MULTICHANNEL ANALYZERS. This one has representatives from Nuclear Data, Nuclear-Chicago, Baird-Atomic, Packard, Victoreen and Picker-Nuclear. The multichannel analyzer, being a basic system for nuclear measurement, it is important that not only the instruments themselves be standardized insofar as practical but that advertising literature and test procedures be based on commonly understood definitions and terminology. Therefore, standards are being developed in areas such as integral and differential linearity and related testing procedures. Other matters for which standards are being developed are in test procedures for stability, count rate dependence, live time accuracy, dead time and gain stability. The type of pulse used for test and the test instrumentation itself are being included in this work.

The next subcommittee is on LIQUID SCINTILLATION COUNT-This has representatives from Packard, Picker-Nuclear, ING. LFE/Tracerlab, Beckman and Nuclear-Chicago. The basic problem they have been working on to date is the establishment of uniform standard samples that can be used in any instrument for checking its performance from day to day and for making comparisons with other instruments. This is not a primary standard or even what you'd call a secondary standard, I think, in radiochemical terminology. We start with National Bureau of Standards radioisotopes, or we use them as a reference for other material. Our interest is in the preparation of the material in sample vials of more or less idealized form with standard scintillator solutions, sealed and flushed with an inert gas to obtain the highest possible efficiency with no quenching. We are also concerned with the preparation of quenched series of standard samples because quenching is an important factor in liquid scintillation counting.

### ANIM-Instruments

There is one more subcommittee - MODULAR CONSTRUCTION. This has representatives from nine A.N.I.M. member companies plus seven non-A.N.I.M. member companies, because the number of organizations involved in the production of NIM Bins and modules is now so large and we want complete representation to present the industry point of view accurately. The work of this group at the present time is essentially keeping in touch with the AEC program.

That pretty well concludes our work and plans to date. I would like to solicit contact with other groups interested in standards to avoid duplication of efforts. We don't wish to attempt to become a real standards producing group. Big formal programs are not presently considered to be within the scope of activities pertinent to our work in A.N.I.M. We currently have only certain limited interests in standards for safety, precision, reliability, and some interchangeability without unduly limiting the designer's freedom to innovate and create new and better nuclear instruments. We further want uniformity in stating specifications and performance, or sort of a truth-in-selling program.

There is just one further item - regarding the use of the metric system. We, of course, can't begin to switch over to metric unilaterally in this country for the entire construc tion of our instruments. However, we are endeavoring to standardize on the use of the metric system wherever possible for dimensions directly affecting the instrument users such as sample sizes, chart speeds, etc.

### STANDARDS NEEDS FOR ACTIVATION ANALYSIS Vincent P. Guinn

The particular requirements of the activation analysis method, in the area of standards, depend upon many factors -one of which is the type of activation analysis. Most of the discussion below will be with reference to the most widely used, and generally most powerful and most applicable type of activation analysis: high-flux thermal-neutron activation analysis (NAA). Lesser attention will be devoted to the other types of activation analysis: NAA with fast neutrons, photonuclear activation analysis, and charged-particle activation analysis.

### THERMAL-NEUTRON ACTIVATION ANALYSIS

In this method of elemental analysis, a sample is exposed to a flux of thermal neutrons for an appropriate period of time, then -- in most instances -- counted on a gamma-ray spectrometer after some appropriate decay period. The technique normally employed is a comparator technique, i.e., a technique in which the counting rate of a particular induced radionuclide activity of interest (or the rates of several activities of interest) is compared with that of a standard sample of the element (or elements) of interest, usually under identical irradiation and counting conditions. Matrix Effects:

Compared with some methods of elemental analysis, thermal-neutron activation analysis, if done properly, is relatively free from matrix effects -- at least for a rather wide variety of kinds of samples. However, it is by no means completely free of matrix effects. Especially where large samples (>1 gram in most cases, >10-100 mg in some cases) are employed, one must consider whether the sample and standard are sufficiently similar in their (1) thermal-neutron absorption (2) epithermal-neutron moderation, and (3) gamma-ray attenuation (absorption and scattering) properties so that no corrections for these factors are necessary. In many instan-175

### Activation Analysis

ces, simple aqueous standard solutions are quite satisfactory. In other instances, however, there is a sufficient mis-match in one or more of these three properties -- between sample and In many cases, standard -- that some action must be taken. the difficulty can be made negligible by simply employing a smaller sample size (and a correspondingly smaller volume of aqueous standard solution). If this is done, however, some sacrifice is made in concentration sensitivity. In other cases, one may choose to match matrices by spiking a portion of the sample with a small volume of the aqueous standard solution -- this standard sample then being compared with the unspiked sample. A third approach -- applicable where the corrections necessary are fairly small -- involves a mathematical correction that is based upon the known or measured properties of the sample, relative to its thermal-neutron absorption, and/or its epithermal-neutron moderation, and/or its gamma-ray attenuation characteristics, compared with the properties of the aqueous standard solution.

In order for various laboratories to establish to their own satisfaction and to the satisfaction of others, that their thermal-neutron activation analysis techniques can properly handle samples that pose problems of the above natures, there is a need for standards that exhibit a range of thermalneutron absorption, epithermal-neutron moderation, and gammaray attenuation properties. If the techniques used are inadequate, somewhat erroneous results will be obtained -- thus drawing attention to the need for improved techniques. If quite good agreement is obtained with the "best" values for one or more elements in such special standards, one can be reasonably confident that the techniques employed are proper for handling even such difficult samples. In such standards, the one or more elements present that are readily determinable by activation analysis with thermal neutrons can be at major, minor, or trace-constituent levels -- since these matrix effects apply to all constituent levels. One has a wide choice of matrices that cover a considerable range in each of

these three properties. Fast-Neutron Interferences:

Another type of standard that can be of real use in NAA work with thermal neutrons is the type that poses special problems in fast-neutron interferences. In most NAA work with thermal neutrons, an appreciable flux of fast neutrons is also present. There are a number of instances, of practical interest, in which fast-neutron activation of one element (one or two units greater in atomic number, Z) produces the same radionuclide that is produced by thermal-neutron activation of the element of interest. Unless this situation is recognized, and either corrected for or virtually eliminated, one can obtain an erroneously high result. A good example is that of determining a trace level of manganese in iron, or in a matrix that is rich in iron. One normally would determine manganese via its thermal-neutron product, 2.576-hour <sup>56</sup>Mn, formed by the  $55_{Mn}(n,\gamma)^{56}$ Mn reaction. However, with fast neutrons, iron can also form <sup>56</sup>Mn: via the  ${}^{56}$ Fe(n,p) ${}^{56}$ Mn reaction. In the reactor sample-irradiation position normally used in the author's laboratory, for example, completely manganesefree iron produces a <sup>56</sup>Mn photopeak counting rate of about  $1.2 \times 10^7$  cpm/g of Fe (in a l-hour irradiation), whereas, under the same conditions, manganese produces a <sup>56</sup>Mn photopeak counting rate of about 2.7 x 10<sup>11</sup> cpm/g of Mn. Thus, uncorrected, really manganese-free iron would appear to contain about 45 ppm manganese. There are quite a few examples of fast-neutron (n,p) and  $(n,\alpha)$  interferences of Z + 1 and Z + 2 elements respectively, with the thermal-neutron  $(n,\gamma)$ determination of the element of interest. There are standard ways (using cadmium shielding of the sample and simultaneous equations) to correct for fast-neutron interferences -- but one must always be on guard to note the possibility of such interferences -- so that one does not fail to make the measurements and corrections that are necessary to avoid an erroneous result. Representative standards that pose such special fast-neutron interference problems can be very helpful, as they can allow a laboratory to determine whether or not it is recognizing such cases of interference, and whether or not their means of correcting for such interferences are sufficiently accurate. Trace Concentrations:

A third general type of standard that is useful in NAA work is the type that contains one or more trace elements at very low concentration levels: for example, in the ppb to ppm range. High-flux thermal-NAA is frequently employed as an analytical technique because of its extreme sensitivity for the detection and quantitative determination of a large number of elements. For example, under the rather typical conditions of a 1-hour nuclear reactor irradiation at a thermal-neutron flux of  $10^{13}$  n/cm<sup>2</sup>-sec, the defined limits of detection for 75 of the elements of the periodic system range from as low as  $10^{-7}\mu g$ (for a few ultra-sensitive elements), to a median of about  $10^{-3}\mu g$ , to as high as  $10\mu g$  (for a few rather insensitive elements). At very low concentration levels, some sources of error that are negligible at higher concentration levels become significant. Container impurities and container recoil effects may be mentioned in this connection. In much NAA work, small vials of rather pure polyethylene are used as irradiation containers for the samples to be analyzed. Although relatively quite pure, the polyethylene does contain ∿ppm and higher concentrations of a number of impurity elements (e.g., 0, Na, Cl, Ti, Al, Mn, Ag, Au). If the activated sample is not transferred to a fresh container prior to counting, and a low level of one of the elements that is present as an impurity in the polyethylene is to be determined in the sample, an erroneously high result is obviously possible. Normally in such cases, of course, one does transfer the activated sample to a fresh container before counting. In general, this eliminates errors arising from container impurity elements -- for sample levels of ~1 ppm and higher. However, when sample constituents present at the ppb level are being measured, even transfer to a fresh container may not eliminate

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the error completely -- because of recoil effects. If the element of interest is present in the container material at ppm levels,  $(n,\gamma)$  recoils will drive a small fraction of the activity induced in the container material into the sample. This can in some cases produce an apparent ppb level of the element in the sample. This container recoil blank can be measured, and applied as a correction. Suitable trace-level standard samples can provide a good means for a laboratory to ascertain whether its procedures are capable of giving accurate values for element concentrations in the sub-ppm region.

#### Radiolysis Effects:

A fourth type of standard that is useful in NAA work is the type that exhibits pronounced radiolysis effects during exposure to neutron, or neutron-plus-gamma, radiation. In thermal-neutron work, this means standards that contain accurately known levels of one or more elements that can readily form, under irradiation conditions, an induced activity of that element appreciably in a volatile form. Elements of particular concern in this connection are the halogens, selenium, and mercury. Due to hot-atom chemical reactions, following  $(n, \gamma)$  recoil, an appreciable fraction of the induced activity can end up in the gas phase of the sample container, rather than in the sample itself, and can be lost (unless special precautions are taken) if the activated sample is transferred to a fresh container. Some of these (particularly mercury) can also diffuse into the polytheylene of the container, and thus again be lost in the transfer process. Such radiolysis effects depend, in their magnitude, not only upon the particular element and the irradiation conditions, but also upon the matrix. In prolonged irradiations at high neutron fluxes, an appreciable amount of some evolved gaseous induced species can diffuse all the way through the polytheylene wall of the container, and hence be lost completely. In such cases, one normally turns to a more impervious, more radiation-resistant, container material, such as aluminum or quartz of very high

### purity.

The above general types of special standards, of particular importance in thermal-neutron activation analysis work, represent the principal ones of value in this field of work. Needless to say, any such standards must be available in a very homogeneous form, since even the very small samples sometimes used in such work must agree rather closely in composition with the gross composition of the material.

### ACTIVATION ANALYSIS WITH FAST NEUTRONS

NAA with fast neutrons is usually conducted with more modest fluxes of neutrons, in the energy range of about 1 -15 Mev. Because of the somewhat lower available fluxes, and the generally much lower cross sections of fast-neutron reactions -- compared with those of most thermal-neutron reactions -- this form of NAA provides, with only a few exceptions, less sensitivity than does high-flux thermal-neutron activation analysis. Thus, in this type of NAA work, there is less need for sub-ppm standards. In practice, because of the generally lower fluxes and generally shorter irradiation times (due to tritium target lifetime limitations, if accelerator-produced 14 Mev neutrons are employed), radiolysis effects are also of less importance. Also, of course, the problem of thermalneutron self shielding is not involved. The usual kinds of fast-neutron reactions of interest and use, in this neutron energy range, are the (n,n'), (n,p),  $(n,\alpha)$ , and (n,2n)reactions. The most common sources of appreciable fluxes of fast neutrons are the nuclear reactor (fission spectrum), the deuteron accelerator (14 Mev neutrons from the (d,t) reaction), and -- soon to be readily available --  $^{252}$ Cf (fission spectrum, from spontaneous fission).

Because of the generally poorer sensitivities attainable with fast neutrons, one frequently endeavors to improve concentration sensitivity by employing larger samples (sometimes even as large as 100 grams). Matrix effects, due to fast-neutron moderation and gamma-ray attenuation, can then

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be appreciable. Standards containing known concentrations of one or more of the elements frequently determined by activation with fast neutrons (e.g., N, O, Si, P, Cr, Fe), in matrices that cover a range of values in their fast-neutron scattering and gamma-ray attenuation properties, can thus be of value.

One very important application of NAA with fast neutrons is the rapid, nondestructive determination of oxygen, using 14 Mev neutrons. Oxygen produces 7.14-second <sup>16</sup>N, via the  $16_{O(n,p)}$  N reaction, this reaction having an energy threshold of 10.2 Mev. The only directly interfering element is fluorine, which forms the same product, via the  $^{19}F(n,\alpha)^{16}N$ reaction. This reaction has a lower threshold: 1.56 Mev. Standards containing known amounts of 0 and F would be of use, to ascertain whether -- via measurement of the product of either the  $19_{F(n,2n)}$  reaction or the  $19_{F(n,p)}$  reaction -- suitably accurate corrections can be made to the observed  $^{16}$ N activity level (in samples containing both O and F) to obtain accurate oxygen values. Boron can also interfere with the 14 Mev-neutron determination of oxygen, since it produces a product (13.6-second <sup>11</sup>Be, via the  ${}^{11}B(n,p){}^{11}Be$  reaction) that emits gamma rays in the same energy range as those of <sup>16</sup>N (6.13 Mev and 7.11 Mev). Thus, standards containing known amounts of both B and O could be of value in checking the reliability of fast neutron oxygen determinations in the presence of a considerable amount of boron (in this case, a resolution of the gamma-ray counting rates obtained at various decay times, in the 4.5 - 7.5 Mev energy range, into the 7.14-second and 13.6-second components, is usually carried out).

Another standard of practical interest could be one containing known levels of nitrogen and copper, since, with 14 Mev neutrons, both elements form a pure positron emitter, with very similar half lives: 9.96-minute  ${}^{13}N(via$  the  ${}^{14}N$  $(n, 2n){}^{13}N$  reaction), and 9.76-minute  ${}^{62}Cu(via$  the  ${}^{63}Cu(n,$  $2n){}^{62}Cu$  reaction). Activation analysis with 14 Mev neutrons is a very promising method for the determination of nitrogen

### Activation Analysis

in foodstuffs (at levels above about 0.1%), but copper, in particular, can in some instances cause results that are appreciably high -- unless a correction is applied.

## PHOTONUCLEAR ACTIVATION ANALYSIS

As yet, this type of activation analysis is not very widely used, nor as extensively developed as NAA, but it should gradually find increasing use. In the photon energy range up to about 25 Mev, the most prominent nuclear reaction, with most elements, is the  $(\gamma, n)$  reaction. Cross sections are generally much lower than thermal-neutron  $(n, \gamma)$ reaction cross sections, with some exceptions. However, in spite of the generally lower cross sections, fairly good sensitivities are attainable for many elements -- if a high photon flux, such as the bremsstrahlung flux produced by a high electron-beam power linear accelerator, is employed. Of particular interest is the possibility of sensitively determining the elements, C, N, and O, via  $(\gamma, n)$  reaction. These three elements form, respectively, 20.3-minute <sup>11</sup>C, 9.96-minute <sup>13</sup>N, and 2.05-minute <sup>15</sup>0. Unfortunately, in many kinds of samples (e.g., metals), these three elements occur together -- and each of these induced activities is a pure positron emitter. If a purely-instrumental analysis is to be performed, one must therefore resolve the 0.511 Mev positron-annihilation photopeak counting rates, observed at various decay times, into the contributions from these three principal species of different half lives. Standards containing known levels of these three elements could thus be of real value. As mentioned earlier, in connection with fast-neutron reactions, copper can also be a contributor to be considered, since it forms the same pure positron emitter, 9.76-minute  $^{62}$ Cu, by (y,n) reaction as it does by (n,2n) reaction.

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### CHARGED-PARTICLE ACTIVATION ANALYSIS

This form of activation analysis is even less developed, less used, and less generally useful than either NAA or photonuclear activation analysis. However, it does have some applications that are of definite potential importance -- predominantly in the area of determining some of the low atomic number elements (e.g., C, N, 0) in medium- to high- Z matrices. Elements in a sample are usually made radioactive by bombarding the sample with energetic (usually 10 - 30 Mev) protons, deuterons, <sup>3</sup>He ions, or alpha particles), employing a cyclotron.

A major problem in standards exists with this type of activation analysis -- one arising from the very limited range of such heavy charged particles in solid or liquid matrices, the rapid slowing down of these incident particles in such matrices, and the energy-dependeces of the various nuclear reactions produced. When such particles enter a sample, the cross section for a particular nuclear reaction of interest may first rise, then decline, and then fall to zero. The excitation function for that reaction depicts the cross section -- versus -- particle energy relationship. The range of the particle will depend upon the type of particle, its initial energy, and the stopping power of the matrix material. However, the range of significant activation can be appreciably less than the actual particle range in any given matrix -- because of the Q value for the reaction (if it is an endoergic reaction), and the Coulomb barrier energy. The effective activation range of the same particle, at the same initial energy, in the same matrix, will be different for a different nuclear reaction -- because it will exhibit a different Q value and/or Coulomb barrier value. At present approximate methods for allowing for differences in stopping power between sample and standard have been developed, but -for really quantitative analysis -- one really needs a standard that is almost identical in stopping power (i.e., in major

### Activation Analysis

composition) with the sample. In some applications of interest, it is possible to use standards that meet this quantitative criterion: discs of selected metals (Fe, Cu, Ti, Al, Be, certain alloys, etc.) that contain known levels of the elements of interest (e.g., C, N, O). In most charged-particle activation analysis work, it is either necessary or at least desirable to have the sample in the form of a thin disc -since one must cool the sample on the back side (to dissipate the heat absorbed in the sample from the beam) and expose the front side to the beam in the high vacuum. Standards should be prepared with this restriction in mind.

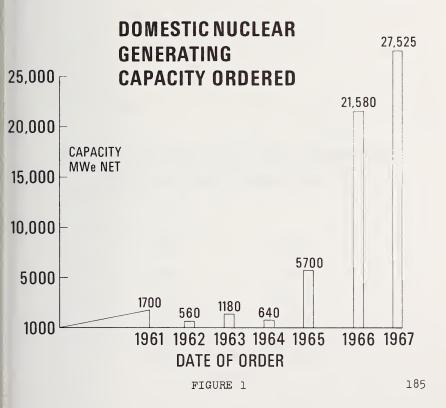
### SUMMARY

Some of the special standards problems, particularly characteristic of the activiation analysis method, in its various forms (thermal-neutron, fast-neutron, photonuclear, and charged-particle), have been reviewed. Particular attention has been devoted to the types of standards useful in high-flux thermal-neutron activation analysis, since this is the most widely used, most fully developed, and most generally useful form of the method. General types of useful standards have been described -- from which various specific ones could be itemized and prepared. Without special standards, activation analysis can still give results of good precision and accuracy, in many instances. However, in many other instances, errors can be made unless suitable precautions are taken and suitable corrections made. Standards that provide good examples of these sources avoiding or correcting for such errors, can be of real value in the field of activation analysis.

## STANDARDS NEEDS OF THE NUCLEAR POWER INDUSTRY J. C. R. Kelly

The nuclear power industry has been growing at an extremely rapid pace. The number of plants is increasing very rapidly. The unit component size of each currently committed plant is eight times the size of the first. The rate of growth will continue. Figure 1 shows the domestic nuclear generating capacity ordered by year.

The output per plant has been increased. The Yankee Rowe plant has four loops with an output of 98 Mwt per loop. A single loop in current plants has twice the capacity of all four Yankee Rowe Loops combined. Units are now offered at 1100 MWe and plants of 1500-2000 MWe are being considered.



This very rapid increase in both the size and number of plants is significant with respect to demands for component and fuel performance and to developing sound and definitive design, production and quality standards.

There are many channels through which the nuclear industr participates in standard preparation. Figure 2 lists a number of them. ORNL-NSIC-43 identifies areas for which standards are needed. These are tabulated in Figure 3 and the status of thos in progress is shown by Figure 4. It will be noted that of the 108 in preparation, only 28 have been approved. Work on standards is not proceeding at a pace compatible with the rate of industry growth. Some ANS standards have been in preparation for 7 or 8 years. The needs of the industry have been identified by industry surveys. Dr. McKune, then vice president of General Electric and now head of USASI, made a survey of industry needs which is summarized in Figure 5 and USASI has reorgan ized from the old ASA to better identify, set priorities, and get sponsors for standards development.

The industry needs are well known at major standards committee levels and at the engineer and scientest level. What are these needs?

# CHANNELS FOR NUCLEAR INDUSTRY STANDARDS PARTICIPATION

ANS	AWS
AIF	ASTM
ASME	ISA
IEEE	USASI
ASCE	ACI

FIGURE 2

# STANDARDS IDENTIFICATION

# ORNL - NSIC - 43 COMPILATION OF UNITED STATES NUCLEAR STANDARDS

# **IMPORTANT EXAMPLES**

SAFETY CRITERIA INSTRUMENTATION AND CONTROL ELECTRICAL MECHANICAL DESIGN CODES FIGURE 3

# **STANDARDS STATUS**

# ORNL - NSIC - 43 COMPILATION OF UNITED STATES NUCLEAR STANDARDS

TECHNICAL ORGANIZATION	ANS	ASME	IEEE	AIF	USASI
STANDARDS LISTED FOR PREPARATION	63	9	14	19	108
APPROVED	5	2	3	0	28
ACTIVE	29	9	11	19	58
INACTIVE	29	0	0	0	22

# **18% HAVE APPROVED STATUS**

FIGURE 4

# NUCLEAR POWER'S STANDARD NEEDS

1. STANDARDS DEVELOPMENT ON SHORTER SCHEDULES

# 2. RECOGNITION AND UTILIZATION OF PRESENTLY APPROVED STANDARDS

3. MAJOR INDUSTRY EFFORT TO EXPEDITE STANDARDS BASED ON REALISTIC CRITERIA

# 4. CONFORMANCE TO EXISTING STANDARDS WHEN THEY ARE SPECIFIED

FIGURE 5

The nuclear power industry needs:

(1) Standards developed on much shorter schedules compatible with the pace of development of the industry.

(2) Recognition and utilization of existing standards in plants, equipment and processes to give the standards recognition and to give users the opportunity to identify weak-

nesses and correct them.

(3) To consider use of draft standards before they are final while they are going through the concensus process in USASI and other organizations. As an example, the ANS has prepared, after several years of work, a standard for Containment Testing Methods. To the best of my knowledge, this has not been referenced by industry in their test plans or by the government in containment test criteria. This document was prepared and reviewed by industry and government and has been available in substantive form for at least three years. (4) A major industry effort to prepare standards on the basis of realistic criteria recognized and utilized by competent people in the field.

(5) To insist on comformance to existing standards. There are many excellent design, material, and process standards that can be and are referenced by industry. Failure to insist on strict conformance to them leads to problems which tend to reflect on the quality of the standards.

There are a number of standards needed which fall in the specific area of the capabilities of chemists. These include both materials and procedures. Figure 6 gives examples.

# STANDARDS NEEDS IN ANALYTICAL CHEMISTRY

# A. MATERIALS

- 1. PLUTONIUM ISOTOPIC STANDARDS
- 2. BURNUP MEASUREMENT STANDARDS OF URANIUM PLUTONIUM - NEODYMIUM
- 3. VANADIUM ALLOY STANDARDS
- 4. ULTRA HIGH PURITY SODIUM STANDARDS

# **B. PROCEDURES**

- **1. BURNUP MEASUREMENT METHODS**
- 2. ANALYSIS OF SODIUM FOR OXYGEN, CARBON AND HYDROGEN
- 3. REVIEW AND UPDATE TID-7029 "SELECTED MEASUREMENT METHODS FOR PLUTONIUM AND URANIUM IN THE NUCLEAR FUEL CYCLE"

FIGURE 6



## NUCLEAR INDUSTRY RESPONSIBILITY IN THE STANDARDS AREA Edwin A. Wiggin

First, I would like to commend the Division of Nuclear Chemistry and Technology and more particularly our chairman, Dr. Meinke, and his associate, Mr. Beeghly, for conceiving of the idea for these sessions and for the comprehensive job they have done in organizing them.

I could not agree more with our chairman when he points out that heretofore there appears to have been little effective communication between the comparatively large number of organizations and groups engaged in the development of nuclear standards. In my opinion, the situation has improved considerably during the past year, largely through the efforts of people like John Landis who is currently serving as chairman of the Nuclear Standards Board of USASI, but I think John would be the first to agree that there is ample room for further improvement.

The Atomic Industrial Forum has been active in the nuclear standards area from the outset, having served as the sponsor of the original ASA N2 and N7 sectional committees. It continues to serve as the sponsor of the successor USASI committees, N12 and N13, respectively. As those of you who attended yesterday's panel sessions may be aware, the N12 committee and its predecessor, N2, have long been engaged in the development of standard terminology and also have taken a leadership role in the ISO work in this area. This admittedly may not be the most exciting type of assignment, but I can think of few other aspects of the nuclear standards program that are more fundamental or important.

The N13 committee and its predecessor, N7, are perhaps best known for their work in developing a standard for the protection of underground uranium miners against radon daughters. The fact that this group had developed a standard, approved as an American standard in 1960, stood the industry in good stead a year ago when the question of the protection of underground uranium miners was publicly raised by the Secretary of Labor and the matter aired at length in public hearings of the Joint Committee on Atomic Energy. The committee is now in the process of developing supplemental standards on air sampling and record keeping to meet priority needs in the aftermath of the hearings.

One additional point that I might make concerning the work of the N13 committee is that it has been appreciably enhanced by the fact that the forum has an active committee of uranium mine and mill operators with which the standards committee has been in continuing contact.

At this point, I would like to turn my attention to a more general discussion of standards activities. It may appear anomalous to be raising philosophical questions about standards development this late in the program. However, on the premise that it is never too late, I take the position that there must be a philosophical rationale for any agreement on which the nuclear industry's responsibilities should be in standards development.

Let me make clear that most of my remarks are directed to those standards relating to the design, construction and operation of nuclear power reactors and especially to those standards bearing on the safety of nuclear power reactors. They might, however, apply to other areas as for example to the safety design and operation of fuel reprocessing facilities. The development of nuclear standards is for a variety of reasons different from the development of other industrial standards. But the development of nuclear power reactor standards is also different from the development of other nuclear standards, as for example those described earlier in this session which relate to isotopes, nuclear instruments, activation analysis and other analytical techniques and measurements. To put it another way, the development of standards for a comparatively small number of multi-million dollar

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machines that are to be designed and built on the basis of still emerging technologies and are to be operated under strict licensing restrictions just has to be different from the development of standards for a line of what by comparison are almost consumer products, the mass production or manufacture of which is based on well established technologies.

Another problem contributing to the complexity of the situation is the problem of semantics. What do we mean by the word, "standard"? Is it the same and interchangeable with the words, "code" and "criteria"? I guess I don't think so. It would also be my guess that any reactor applicant who has been through the AEC licensing review process and has been required to explain how the pressure vessel for his unit will conform to the AEC's supplemental criteria would agree. Although I am not prepared to come up with a rigorous definition of each, I would submit that this is perhaps a problem that should be referred to N12.

There is still another facet of nuclear standards development which at least historically sets it apart from the development of other industrial standards. The development of most industrial standards has been based on the identification and resolution of an existing problem. To reach back into history for a simple example, I am told that when electrical appliances first came on the market, there was no uniformity between the appliance plug and the wall outlet into which it was supposed to fit. A more current case is the noninterchangeability of cassettes for cartridge tape players. The need for a standard in both instances is obvious. Generally speaking, once a problem has been solved and the solution proved valid by experience, whether the solution is a modified design or a new analytical technique or an improved fabrication practice, it is codified as a standard. Its acceptance by industry, and for that matter by government, is usually pro forma since the need for the usefulness of the standard has already been well documented by experience.

### AIF-Industry Responsibilities

The situation in the nuclear standards area has been and still is quite different, primarily because the need in most instances is far less obvious. Also, comparatively speaking, the technology is changing much more rapidly. When the ASA first set up the Nuclear Standards Board in 1956, it was difficult to determine what the real problems would be, much less identify those which would lend themselves to solution by the development of a standard. There was little experience in designing and constructing power reactors and no operating experience. Indeed, a nuclear industry, as such, had hardly come into existence. Apparently, those who were instrumental in establishing the Nuclear Standards Board believed that nuclear standards could be developed as prophylactic measures - a concept to which I personally do not subscribe. The fact that the USASI catalog lists only 15 nuclear standards approved during the first 12 years of the program despite countless man hours of meetings and drafting effort appears to add credence to my argument. I might also note that only one of the 15 relates directly to the design, construction and operation of nuclear power reactors.

In rebuttal to those who might be prone to point out that this is ancient history and therefore not germane to the question of where do we go from here, I would say that like it or not it tends to dictate as well as circumscribe industry's nuclear standards efforts for the immediate future.

I think it fair to say that up until a year or so ago the nuclear standards program had achieved a record of much activity and limited accomplishment. Let me hasten to add that I am not critical of such a record of accomplishment in an area in which there has been little need and little desirability for standards. But the past level of non-productive activity has been unfortunate inasmuch as it has prompted the AEC to take a position that in absence of any evidence of an industrial capability to develop standards it had little alternative other than to take the initiative and do the job itself. By way of example, I cite AEC's "Tentative Regulatory

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Supplementary Criteria for ASME Code-Constructed Nuclear Pressure Vessels" and its preliminary unpublished efforts to develop standards or criteria for in-service inspection of the primary systems of nuclear power reactors and quality control standards for field fabrication and construction work.

Without intending to belabor the inadequacy of such a unilateral government approach, suffice it to say that in the opinion of an ad hoc committee of pressure vessel suppliers and users convened by the Forum to review the supplementary pressure vessel criteria, only seven of some 62 sections set forth safety criteria requirements of a type which would appear appropriate to include in AEC licensing criteria. Of the balance, 52 referred to code type requirements which the committee suggested should be referred to the appropriate industrial code writing group, two referred to engineering procurement and design specification type requirements and one was considered to be an owner-purchaser responsibility. The committee further found that some or all of the requirements set forth in five of the sections could not be met on the basis of current technology.

Since the first of this year, a number of changes have taken place which would lead me to hope that we will not see a repeat of this type of unilateral effort. The Nuclear Standards Board has been successful in soliciting AEC participation in a number of its working committees and AEC representation on its Executive Committee. Also, the NSB Executive Committee has apparently recognized that it must take a much more positive approach in recommending what standards work needs priority attention by what working group.

Hence at long last, it would appear that industry has a chance of regaining the initiative and this is as it should be.

### AIF-Industry Responsibilities

Earlier this morning, you heard Mr. Crawford describe the AEC's future needs for standards in reactor development and technology. Some of those needs are undoubtedly the same as the future needs of the commercial nuclear power industry. However, as we have told the AEC, it would be ill advised to assume that the requirements which the AEC may find reason to write into its procurement specifications should necessarily turn up in the regulatory criteria to be used by the AEC in reviewing license applications. When this does happen, the industry for all intents and purposes is faced with a fait accompli requirement to come up with some kind of a standard, not because it is necessary or even desirable but simply to satisfy an arbitrary licensing requirement which may have little or no relevancy to health and safety.

To sum up, my remarks are intended as a warning flag. The industry must maintain the initiative in standards writing. It must continue to seek the active participation and concurrence of the AEC and other involved government agencies because reactor construction and operation will by necessity of sound public policy as stated in the law remain a strictly licensed activity. I don't think any responsible representative of the nuclear industry questions either the need or the desirability of this prerequisite.

But the industry must also be satisfied that there is a need for a particular standard and that it will serve to advance nuclear power technology rather than retard it, or even worse set it back. Further, it must agree to abide by the standard once approved and assure itself that the AEC and such other licensing authorities as may be involved are willing to accept the standard as satisfying or being compatible with a license requirement.

These may not prove easy objectives to attain. I submit, however, that three steps industry can take in an effort to seek attainment are: (1) to develop, where possible, "performance" standards in contrast to "specification" standards, (2) to seek the increased active participation of user repre-

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sentatives, particularly the utility people, in the development of standards, and (3) to carefully document its operating experience in order that appropriate modification of initially established standards can be made in a timely fashion.



## THE NATIONAL STANDARD REFERENCE DATA SYSTEM \*

Edward L. Brady and Merrill B. Wallenstein

Improvement in the effectiveness of the nation's system for scientific and technical information is a matter of great popular concern these days. Much is being said and written about the flow of information from the generator to the user, and much is being done to try to speed the process. Taking the broadest possible approach, the President's Office of Science and Technology is examining all aspects of the problem [1].

The Chemical Abstracts Service of the American Chemical Society is in the midst of a long-range program designed to increase the retrievability of information within its concern [2]. Similarly, the American Institute of Physics had embarked on a comprehensive study of means to make the world's output of information in physics more readily available to individual users [3]; the Engineers Joint Council has a similar program [4]. The Atomic Energy Commission, the Department of Defense, the National Aeronautics and Space Administration, and other major federal technical agencies are all increasing their efforts to improve the use of information generated within their programs.

These government activities are coordinated through the Federal Council for Science and Technology by means of its Committee on Scientific and Technical Information (COSA TI), consisting of representatives of all government departments and independent agencies that have major technicalinformation programs. It was the initiative of COSATI and its parent council that led to establishment in 1963 of the National Standard Reference Data System, a federal interagency activity concerned with one aspect of the broad problem of scientific and technical information--improvement of

\* Reproduced from SCIENCE, May 12, 1967, Vol. 156, 3776, p.754.

#### Reference Data

access by the American technical community to compilations of critically evaluated data on the properties of substances.

Such compilations have been among the basic tools of scientists and engineers throughout the history of technology; each owns at least one handbook containing, among other useful information, table after table of data on the properties of the substances and systems that he deals with daily. Systematic compilations of data also contribute in a fundamental way to progress at the forefront of science. Samuel Goudsmit [5] recently emphasized this importance with the following words:

Experimental results in measurements are the backbone of physics. No theory is acceptable unless it agrees with the experimental data. Conversely, a systematic study of experimental results can suggest new theoretical approaches. Tables and graphs of numerical data therefore play an important role in the progress of physics. . . . It is thus obvious that specialized data compilations are of great importance and should have the full cooperation of those producing the data. It is also clear that modern computer techniques can handle such data more efficiently than old tabulations could, especially since their number and variety are growing so rapidly.

Since the numerical data that result from measurements of properties normally appear somewhere in the world's literature, why not let the individual scientist or engineer look them up whenever he needs a value? There are two major reasons why this procedure is not efficient: First, it is often very difficult to locate a desired value among the millions of papers stored in a technical library; searching indexes, abstracts, and papers can consume many hours. Second, conflicting values for the same property are often reported; unless the user is a specialist in the field, he will have difficulty in deciding which value he should use. These inefficiencies translate directly into money. If the average

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scientist or engineer were to save only 10 minutes a week that he now spends finding and evaluating data, the annual saving to the nation's research and development program would be of the order of \$100 million. This estimate takes no account of the benefits of having better data, evaluated by an expert in the field, at hand when needed. Obviously, very significant economic benefits can be readily gained by organizing a coordinated, comprehensive program for reviewing the literature, extracting and evaluating the property data contained therein, and disseminating them in convenient form.

Because of their usefulness and economic benefits, many compilations of data [6] have been produced throughout the world, largely in response to urgent needs of the technical community. However, existing mechanisms have not been able to keep pace with the flood of new data appearing in the literature, except in a few specialized areas. Some compilations were "one shot" projects, resulting in products that were never updated; others have been continuing activities lasting many years. Some have been sponsored by missionoriented agencies of the United States government; others, by private organizations. However, many newly recognized properties are not covered at all, and the time lag between the appearance of data in original literature and their evaluation for inclusion in a critical compilation has been rapidly increasing. Moreover, even in the areas covered by active projects there was little coordination or standardization of format or quality, and in some technical areas there was extensive duplication. The National Academy of Sciences, which in earlier years had been responsible for production of the widely used International Critical Tables, made an important contribution to coordination and stimulation through its Office of Critical Tables, but this office has neither directive nor resources to manage an operational program.

Recognizing the deficiencies of the existing situation and the stake of the U.S. government in the financial support

of the nation's research and development activity, COSATI decided that a government-wide coordinated effort was needed; thereupon it recommended that a proposed plan of action for increasing the level of effort of the National Bureau of Standards in this field be expanded to encompass the total federal effort within all agencies, with administrative responsibility assigned to the Bureau. Adopting this recommendation, the Federal Council for Science and Technology and the President's Office of Science and Technology, then headed by Jerome Wiesner, promulgated a federal policy establishing the National Standard Reference Data System (NSRDS).

The NSRDS is regarded as a sub-system within the concept of the "National Measurement System" [7]. The "National Measurement System" is envisioned as comprising a central core of national standards of measurement, a set of consistent instruments (calibrated through appropriate application of the national standards), a body of reference data that provides users with ready-made answers to questions on the properties of substances, and finally the entire set of meaningful measurements made throughout science, technology, and the economy. From this viewpoint, NSRDS is regarded as a portion of the activities leading to dissemination of ready-made data for use by the technical community of the United States.

# RESPONSIBILITIES OF THE NATIONAL BUREAU OF STANDARDS

In accepting the charge from the Federal Council for Science and Technology, the Bureau has taken responsibility for (i) promoting the general objective by sponsoring critical-evaluation and data-compilation projects as needed, (ii) coordinating related work under the auspices of all government agencies, (iii) establishing standards of quality for products of the system, (iv) operating a national center for standard reference data, and (v) establishing standards of methodology and such other functions as are required to ensure

the compatibility of all operational components of NSRDS. The goals of NSRDS are to be achieved through operation of an integrated network of data-evaluation centers and related projects located wherever special technical competence for a particular project may exist.

Since data can be adequately evaluated only by specialists whose judgments are respected by their peers, each data center is to be concerned with a carefully delimited technical scope; normally it will be established as an adjunct to the work of an individual or group having an established reputation for competence and vigor. This principle of operation was strongly recommended in the report of the Weinberg committee [8], and its importance has been fully demonstrated in NSRDS operations.

As described so far, the technical scope of the standard reference data system undoubtedly appears limitless. However, the Bureau, with the concurrence of the interested federal agencies involved in NSRDS, has endeavored to avoid being cast into the infinite sea of data that exist for scientific and technical properties and substances of all types.

Guidelines have been established to limit the boundaries of NSRDS which, hopefully, restrict its program to a manageable size. The program is to be concerned with (i) the data of physical science only (data relating to biologic phenomena will be excluded); (ii) well-defined substances only (substances whose composition, structure, and energy content are so precisely known that measurements of the property under consideration do not wander erratically); and (iii) only well-defined properties that are intrinsic to the substance or system being studied (properties that must be defined in terms of the system used for measurement--such as Brinell hardness and Charpy breaking strength--are excluded). With these constraints, the task of NSRDS is probably feasible. Because of limitations of manpower and funding resources since the establishment of NSRDS, the task has not yet been

determined to be practical. Although we who are connected with the program sometimes feel overwhelmed by the magnitude of what we are trying to do, wherever we look we see exciting and challenging opportunities to make important contributions to science and technology.

# OPERATION OF THE NATIONAL STANDARD REFERENCE DATA SYSTEM

Within the National Bureau of Standards the responsibility for administering NSRDS has been assigned to the Office of Standard Reference Data, created for that purpose within the Institute of Basic Standards. Three major groups of activities within the Office of Standard Reference Data have been initiated: these are concerned with (i) the planning and implementation of projects for compiling data, organized into several broad technical areas; (ii) an information-systems design and research activity; and (iii) various specialized information services to be provided to the technical community.

For program management the data-compilation projects of the Office of Standard Reference Data have been subdivided into seven broad subprograms: (i) nuclear properties, (ii) atomic and molecular properties, (iii) solidstate properties, (iv) thermodynamic and transport properties, (v) chemical kinetics, (vi) colloid and surface properties, and (vii) mechanical properties. In each, responsibility for developing a comprehensive, coordinated program has been assigned to a program manager.

Existing projects of other governmental and nongovernmental agencies are taken into account, and project priorities are determined by consultation with groups of specialists from the academic world, government, and industry. Some of the projects are conducted within the experimental divisions of the Bureau; others, in university laboratories or in other government laboratories; and a few, by industry. None is under the direct operational supervision of the Office of

Standard Reference Data, which is exclusively for program management.

The level of effort in each project supported by the Office of Standard Reference Data is determined by a practical compromise involving three considerations: (i) the degree of comprehensiveness of the literature review (ii) the procedure for critical evaluation applied to the data, and (iii) the need for continuity in updating the compilations. These three considerations require further discussion.

The raw material for any data-compilation project is the results of measurements by the whole world. Normally, these results are reported in the literature, some in journals which, however, may be obscure or difficult to obtain. Moreover, an increasing fraction of results worth saving for posterity is now appearing in government reports. Furthermore, in some areas (data on neutron cross sections are one example) many of the data generated in the laboratory never appear in any report or publication; in such instances the compiler personally may have to pry the data from the measurer. For a specific case, the degree of comprehensiveness that can be achieved must be a practical compromise between the desired 100 percent and the cost in time, money, and effort of achieving that goal. For most existing projects the comprehensiveness probably attains 90 to 99.8 percent.

The procedure for "critical evaluation" varies widely from project to project. In present practice in some data centers, the experimental technique is reviewed, calculations are spot-checked, values of the fundamental constants are checked to ensure that the latest values are used, the temperature scale is checked (if appropriate), and limits of experimental uncertainty are independently assessed. In other centers, the data evaluator may decide, for intangible reasons that he may find difficult to formulate, that one particular value in the literature is "better" than another value. Such a judgment by a specialist of broad experience should not be

underrated; the value obtained is much more likely to be accurate than the result of unweighted averaging. Most people agree that the first procedure provides a better "critical evaluation" than the second. However, for the practical purposes to which many compilations are applied, such a review is not justified, and the second procedure, or an intermediate one, is employed.

The question immediately arises, then, of what degree of critical evaluation is required for a compilation to be considered "standard" reference data. It is probably desirable to use the word "standard" sparingly; it has connotations that apply to few existing compilations. For the present, when measurement results for most properties are uncertain and many are in dispute, the shorter term "reference data" would avoid the implications aroused by use of the word standard. "Standard reference data," the ultimate goal of NSRDS, are to be striven for constantly, but perhaps not reached in many fields for years. Because of the variation in procedures for critical evaluation, all publications of NSRDS are to describe the criteria used for judgment and the argumentation used to derive the recommended values.

For each individual compilation project, requirements for continuity must be examined. The overall program of NSRDS is designed to ensure continuity of effort in production of data compilations needed by scientists and engineers. In some areas a revised and updated compilation may be needed every 6 months; in others, only every 4 or 5 years. In almost all areas, continuing literature review and indexing operations are required to maintain a current awareness of the state of development of the field. Therefore most new projects undertaken by NSRDS are expected to be longterm, continuing activities, maintained as one component of the normal range of professional activity of the leader of the program.

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The types of activities and products of these data centers will now be examined in some detail. Figure 1 is a schematic diagram of the broad types of activities and products that are normally associated with a data center. The left-hand column represents activities, while the righthand column indicates a product that may result from the corresponding activity on the left. Following the initial selection of relevant papers from the literature (an activity basic to all evaluation and compilation projects), a bibliography may be prepared, in which the literature to be evaluated is classified into several relatively broad categories. After the initial selection, the papers are indexed; this process consists of assigning a number of key words or symbols to each paper to indicate the data content of the reference. The indexed bibliography resulting from this activity is very useful to many groups of specialists.

Figure 2 shows a page from one of the most comprehensive and successful of these indexed bibliographies, called CINDA [9] (Computer Index Neutron Data); it is concerned with sources of data on neutron cross sections. The first several columns contain symbols and numbers representing the target nucleus, the range of incident neutron energy, identification of the reference in a list following the table, and identification of the laboratory at which the measurements

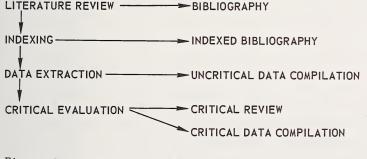


Figure 1. Activities and products of data-evaluation centers.

208		Reference Data
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CHROMIUM No.	500867 9255 9225 9225 9225 9225 9228 9229 584	500868 SAC 586 586 587 587 588 588 588 588 591 593 593 593 593 594 593 595 595
257 ENTRY DATE	6507204 650420EU 650210U 650210U 650210U 650210U	650407EU 650210U 650210U 650210U 650210U 650210U 650210U 650210U 650210U 650210U 650210U 650210U
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Illustrative page from CINDA [9], an indexed bibliography. Figure 2.

were carried out. Next come additional remarks characterizing the measurement more fully, the date of addition of the item to the computer file, identification of the person who prepared the entry, and an accession number for the entry. CINDA is particularly noteworthy because it represents the combined efforts of scientists in the United States, the United Kingdom, France, Germany, the U.S.S.R., and other countries.

In consultations to determine the needs of the technical community of the United States for data compilations of all kinds, the staff of the Office of Standard Reference Data has been told often that an indexed bibliography of this type would satisfy most of the needs of the specialists, since many of them would prefer to evaluate the data themselves and wish only to avoid the labor of locating sources of the information. However, this attitude does not prevail among those who need a particular value for a calculation of some kind and are not themselves involved in research in the field.

The next step in the production of a critical compilation is the extraction of data from the literature that has been selected. At this stage an uncritical compilation could be issued, if determined to be useful to the technical community. Figure 3 is an example of this kind of product: a print-out of data on the ionization and appearance potential of cyanogen ion, retrieved from the files of the Mass Spectrometric Data Center at the National Bureau of Standards in Washington. This material is retrieved as required to satisfy the requests of individual inquirers. A similar product is now being considered by the Nuclear Data Project at Oak Ridge National Laboratory, in response to requests by members of the U. S. nuclear-physics community; it would consist of a reproduction of the raw data extracted from the papers published in the field of nuclear structure. The normal product of the Nuclear Data Project consists of carefully evaluated

# MASS SPECTROMETRIC DATA INFORMATION CENTER-NBS

Listed below is the requested information as obtained from the literature since 1955.

ION REACTANT PRODUCTS IP or AP		YEAR	DOC NO
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	SP SP IB SP SP SP SP SP SP SP SP	60 600 555 61 61 61 62	00073 00073 00090 00090 00154 00154 00154 00154 00154 00154 00154

TERM DESIGNATION

SP=SEMILOG PLOT IB=INITIAL BREAK

EVD=EXTRAPOLATED VOLTAGE DIFFERENCE

#### REFERENCES

00073 Herron, J.T. and Dibeler, V.H., "ELECTRON IMPACT 00073 STUDY OF THE CYANOGEN HALIDES, J.Am. Chem. Soc. 00073 82,1555(1960)

00090 Kandel,R.J., "APPEARANCE POTENTIAL STUDIES.II. 00090 NITROMETHANE", J. Chem. Phys. 23, 84(1955)

00154 Dibeler, V.H., Reese, R.M. and Franklin, J.L., 00154 "MASS SPECTROMETRIC STUDY OF CYANOGEN AND CYANOACETYLENES 00154 J. Am. Chem. Soc. 83, 1813(1961)

00202 Kiser, R.W. and Hobrock, B.G., "THE IONIZATION 00202 POTENTIALS OF CYCLOPROPYL RADICAL AND CYCLOPROPLY CYANIDE 00202 J. Phys. Chem. 66, 957(1962)

Please note that we make no claim that the above information has been critically evaluated by NBS personnel nor do we make any claim that there is a preferred value.

We hope that we may be of further assistance to you in the future

Georgia L. Apostolou Mass Spectrometry Section Institute for Basic Standards National Bureau of Standards Washington, D. C. 20234

Figure 3. Print-out of data from NBS Mass Spectrometric Data Center, typical of uncritical-data compilation with bibliography. energy-level diagrams and other quantitative data, and will continue to appear regularly.

The preliminary activities of literature selection, indexing, and extraction of data lead finally to critical evaluation of the data. The product of this work is a critical view of the state of quantitative knowledge in some limited area of a field, or a compilation of critically evaluated data. For NSRDS, a published product must contain sufficient argumentation for the user of the data to know how the results were obtained, as well as appropriate reference to the sources of the data used in the final evaluation. Figure 4 shows a page from a typical product of this type, with data expressed in the form of a table of numbers; data may also be expressed graphically (Fig. 5).

In accordance with the directives of NSRDS, only activities leading to the production of a critical review, or compilation of critical data, are considered appropriate for support by the Bureau's Office of Standard Reference Data. However, because the intermediate products are often very useful, NSRDS data centers may issue them also, along with other publications.

The physical form of the products of NSRDS activities may be anything considered convenient by the users to whom the product is directed; that is, the product may be a monograph, loose-leaf data sheets, a journal article, microfiche cards, IBM punch cards, punched paper tape, magnetic tape, or any other physical form in which information may be stored.

The information-systems design and research activity of the Bureau's Office of Standard Reference Data is concerned with the problems of handling data throughout the entire flow process; that is, from the time measurements are first made in the laboratory, through disclosure in some form to other persons who may use the results (as a journal article, a laboratory report, or perhaps a magnetic tape), through the review, selection, and evaluation procedures in the data cen-

## Ground State

**Ionization** Potential

1s<sup>2</sup>2s<sup>2</sup>2p <sup>2</sup>P°<sub>1/2</sub>

24.376  $eV = 196659.0 cm^{-1}$ 

## Allowed Transitions

Сп

List of tabulated lines:

Wavelength [Å]	No.	Wavelength [Å]	No.	Wavelength [Å]	No.
687.059	10	3360.9	27	4413.2	24
687.35	10	3361.09	27	4618.9	26
687.355	10	3361.75	27	4628.1	26
858.092	- ğ	3581.80	18	5132.96	14
858.559	9	3584.98	18	5133.29	14
903.624	3	3585.83	18	5137.26	14
903.962	3	3587.68	18	5139.21	14
904.142	3	3588.92	18	5143.49	14
904.480	3	3589.67	18	5145.16	14
1009.85	4	3590.87	18	5151.08	14
1010.07	4	3876.05	22	5640.50	13
1010.37	4	3876.19	22	5648.08	13
1036.34	22	3876.41	22	5662.51	13
1037.02	2	3876.67	22	5889.4	20
1065.88	6	3878.22	22	5889.97	20
1065.9	6	3879.60	22	5891.65	20
1066.12	6	3880.59	22	6578.03	11
1323.9	5	3881.2	22	6582.85	11
1334.53	1	3883.8	22	6779.74	12
1335.66	1	3918.98	17	6780.27	12
1335.71	1	3920.68	17	6783.75	12
2173.8	15	4074.53	23	6787.09	12
2174.1	15	4076.00	23	6791.30	12
2509.11	7	4267.02	21	6798.04	12
2511.71	7	4267.2	21	6800.50	12
2512.03	7	4267.27	21	6812.19	12
2746.50	19	4371.59	25	7231.12	16
2747.3	19	4372.49	25	7236.19	16
2747.31	19	4374.28	25	7236.2	16
2836.71	8	4411.20	24	18895	29
2837.60	8	4411.52	24	18916	29
2992.6	28				

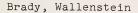
Self-consistent field calculations by Weiss [1], and Biermann and Lübeck [3], and a high current arc experiment by Maecker [2] are utilized for the tabulation. The results for the lower and moderately excited transitions should be quite uncertain because in the calculations the strong effects of configuration interaction are essentially neglected, and the experimental work is subject to large systematic uncertainties.

#### References

Weiss, A. W., private communication (1964).
 Maecker, H., Z. Physik 135, 13-22 (1953).

[3] Biermann, L. and Lübeck, K., Z. Astrophys. 25, 325-339 (1948).

Figure 4. Illustrative page from "Atomic Transition Probabilities" [11], a critically evaluated compilation



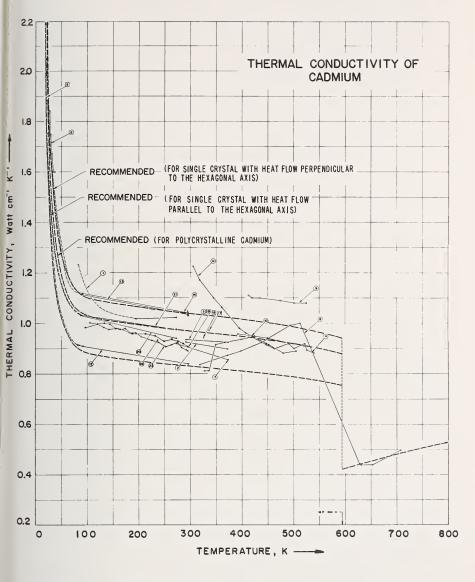


Figure 5. Illustrative page from "Thermal Conductivity of Selected Materials" [12].

ter, and finally to dissemination among members of the technical community who have use for the evaluated product. The use of computers for all practical operations is emphasized; such applications to speed the flow of data from the measurer to the user have only just begun.

Several user services are being planned, utilizing the storehouse of data to be contained in the Bureau's Standard Reference Data Center. This storehouse will eventually contain a complete collection of compilations of critically evaluated data produced throughout the world.

The following list indicates the services that are available or definitely planned as part of the office program:

 Referral: referral of a request for data on a specific subject to a center specializing in that subject.

2) Reference: provision of literature references in response to a request for information, with an indication of where the requester may locate relevant data.

3) Documentation: provision of copies of documents in response to inquiries (perhaps ranging from a Xerox copy of a page to a complete bound volume).

4) Data: provision of detailed data as required to respond fully to a request for information; the service might range from a reply to a question such as "What is the value of property P for substance S at temperature T?" to replies to questions such as "What substances have values for property P in the range c to d, and for property Q in the range f to g, but not for property R in the range j to k?"

5) Current-awareness: periodical or aperiodical announcement of new products and services of NSRDS, describing in some detail the properties, substances, and ranges of parameters covered by compilations, and explaining the means of access to the items described.

At present, services 1 and 5 are active. The Bureau hopes that it will not be swamped with requests for data as

a result of this announcement, because it is not equipped to process many inquiries. The current-awareness service consists of a monthly newsletter sent to persons requesting it; it is now free but soon there may be a small charge.

Products and services still under consideration include:

 Special handbooks: preparation of handbooks containing selected portions of data compilations needed by individuals or mission-oriented segments of the community (such as data needed by oceanographers, upper-atmosphere physicists, or desalination engineers).

2) Format conversion: conversion of data compilations from one physical form to another; for special purposes, a customer may wish to have data on a magnetic tape rather than on a printed page, or on punched cards rather than punched paper tape, or, in general, in some form other than that of the original product.

3) Property computation: computation to special order of experimental properties that may be stored as mathematical relations, or that must be calculated from theoretical or approximation equations.

4) Remote access: making the central bank of stored data accessible to a remote console anywhere in the United States (or conceivably anywhere in the world by way of microwave relay or a communications satellite).

The program decisions of the Bureau's Office of Standard Reference Data have relied heavily on the advice of a representative cross section of the American technical community. As overall program-review committee for the work of the office, the executive committee of the Office of Critical Tables of the National Academy of Sciences-National Research Council provides policy recommendations and is an important channel of communication with many segments of the technical community. Program officers in other government agencies have been consulted to determine the needs of their mission-

oriented programs for the products and services that the standard reference data system is intended to provide. Considerable reliance has been placed on the recommendations of panels of specialists in each of the technical categories in which a program is being operated; one or more meetings of ad hoc panels have been held in each area. Some of these panels were existing committees of the NAS-NRC, established primarily for other purposes; others have been assembled for the purpose directly by the Bureau. These advisory panels are now being organized on a continuing basis under the auspices of the National Academy of Sciences-National Academy of Engineering. The NAS-NRC Office of Critical Tables has provided frequent advice on needs, priorities, and other operational details, and has also served as a channel of communication to segments of the technical community in the United States and abroad that would be difficult to reach in other ways. In all, more than 200 leaders of American science and technology have given generously of their time and experience in helping to make NSRDS most responsive to the needs of the technical community.

## GENERAL STATUS OF PROGRAM

As a result of the recommendations of advisory panels, greatest emphasis has been placed on initiation of new projects for evaluation and compilation and on expansion of old ones, leaving to a future of greater affluence the implementation of extensive and sophisticated information services. Significant progress has been made, especially in the areas of thermodynamic and transport properties and of atomic and molecular properties; these two categories have been judged to be of highest priority for additional effort. In the field of nuclear data, existing activities sponsored by the U. S. Atomic Energy Commission provide nearly adequate coverage of the technical scope required, although the level of effort needs to be increased to meet the rapid rate of appearance of new data. For solid-state properties, existing projects provide good coverage of the more classical areas

(such as structural data), but greatly increased effort on the newer kinds of data (such as energy levels, band structure, and interaction with radiations) has been recommended by the advisory panel. For chemical kinetics, the advisory panel recommended that the first step be preparation of a series of critical reviews on the state of quantitative knowledge in certain selected aspects of the field, since the panel members were not at all certain that any quantitative data in the literature were worth a systematic compilation project.

For colloid and surface properties, the Bureau's Office of Standard Reference Data has established a cooperative relation with the NAS-NRC Committee on Colloid and Surface Chemistry, which had been planning an extensive program of data evaluation before NSRDS was established. For mechanical properties, from a preliminary critical examination, by a panel of Bureau staff members, it was concluded that most results of mechanical-property measurements are unlikely to satisfy criteria for "standard" reference data; this tentative conclusion is to be examined soon by a panel representing a broader selection of specialists from outside the Bureau.

More detail on the activities of the Office of Standard Reference Data and on the status of specific projects under the cognizance of this office are described in a recent report by the Office of Standard Reference Data [10].

### PROPOSED LEGISLATION

During the 3 years of the Bureau's administration of NSRDS, a start has been made toward satisfying the general obligation of supplying reference data to the American technical community. These years have, however, revealed the desirability of additional authority from Congress for increased efficacy. Seeking such authority, the Department of Commerce submitted draft legislation to the 89th Congress. After public hearings before the Daddario Subcommittee on Science, Research, and Development (House Committee on

Science and Astronautics), a revised bill was reported favorably by the full Committee; it was passed by the House of Representatives in mid-August 1966. However, the Senate Commerce Committee, which oversees the program of the Bureau, did not hold hearings or report the bill to the Senate. The bill has been resubmitted for consideration by the 90th Congress.

The legislation, as revised after the hearings of the Daddario subcommittee, contained the following provisions: (i) a declaration that it is the policy of the Congress to make critically evaluated reference data readily available to scientists, engineers, and the general public; (ii) a directive to the Secretary of Commerce to provide or arrange for the collection, compilation, critical evaluation, publication, and dissemination of standard reference data; (iii) a directive to the Secretary of Commerce to prescribe standard criteria and procedures for the preparation and publication of standard reference data, as may be necessary; (iv) authority for the Secretary, or a person or agency designated by him, to sell standard reference data and to allow the proceeds to be used by the Bureau; (v) authority for the Secretary to obtain copyright, on behalf of the United States as author or proprietor, in standard reference data prepared or made available under the Act; and (vi) an authorization for appropriations in such amounts as may be needed for the purpose of the Act.

## INTERNATIONAL COOPERATION

Evaluation and compilation of data on the properties of substances has been a joint activity of the world's scientists for many years. The International Critical Tables, produced mainly between 1920 and 1930, contained contributions from scientists all over the world, coordinated through the efforts of the National Academy of Sciences. The tables of Landolt-Bornstein, originally German, now contain contributions by scientists from many countries. Compilation and evaluation of neutron cross-section data have become a broad

international effort, with participation by centers in the United States, Canada, the United Kingdom, France, the Soviet Union, and other countries.

The establishment of NSRDS in the United States has stimulated additional interest among scientists in other countries in the possibility of developing cooperative programs with scientists in the United States. Possible cooperation has been discussed with scientists from the United Kingdom, France, Germany, the U.S.S.R., and Japan. Such widespread interest leads immediately to the concept of a multilateral international program, incorporating activities from all countries wishing to participate. Indeed, multinational cooperation through several of the international scientific unions has been under way for many years. The International Unions of Pure and Applied Chemistry and Pure and Applied Physics and the International Astronomical Union have been especially active. In June 1966 the International Council of Scientific Unions created a Committee on Data for Science and Technology (now called CODATA) whose function is to coordinate projects for data compilation and stimulate the formation of new ones on an international basis. This committee is served by a small professional staff, headed on a part-time basis by the present director of the Office of Critical Tables of the National Academy of Sciences. For 1 or 2 years the office will be located in Washington, D. C., and then will probably be moved to Europe.

Competence and interest in an international cooperative program for compiling reference data are found in most of the technically developed countries. The products of such a program would benefit any nation that conducts a research and development effort of any size, not merely the most highly developed countries. International cooperation in this area has been a tradition of the world's scientists for at least half a century, but until recently no mechanisms for overall coordination and support have existed. In short, many arguments now favor the vigorous development of an international cooperative program.

There can be no doubt that the computer will ultimately change all practices in obtaining, collecting, evaluating, and transmitting data. On-line computers, coupled with new instrumentation, will increase enormously the rate of measurement of properties and of analysis of experimental data, raising immediate questions of what and how much should be printed in a publication. The processing of data and literature in an evaluation center will be handled largely by computers. Journal articles, monographs, and other printed records will be composed by computer-controlled photocomposition devices. Data and other information will be stored magnetically and will be available to scientists and engineers by way of remote-access consoles. All these developments now exist; they will undoubtedly transform the working habits of scientists and engineers everywhere. However, improvements in the mechanics of processing data can only serve as an aid to the evaluation process, which can be done only by well-trained human brains. For this reason we consider that the basic concept of NSRDS -- establishment of a comprehensive network of centers in which experts evaluate data in their fields of specialty -- has validity for many years to come.

# CONCLUSION: A WORD TO THE TECHNICAL READER

An essential element in the control of any system is feedback. Successful control of NSRDS is impossible without feedback from the members of the technical community, which includes the readers of this article. We need feedback from you in order to determine the priorities of compilation projects, to determine the kinds of services that you need the most, and to judge how successfully the system operates. Right now you, the reader, could let us know, for example, what properties, of what substances, you need to have in the form of a critically evaluated compilation. Let us know if

### Brady, Wallenstein

you yourself are working on a critically evaluated compilation and need help to finish or publish it. Give us any advice you think we need. We seek the cooperation and assistance of the entire technical community in achieving our common goal of promoting the technical advancement of the United States.

#### SUMMARY

The National Standard Reference Data System is a government-wide effort to give to the technical community of the United States optimum access to the quantitative data of physical science, critically evaluated and compiled for con-This program was established in 1963 through acvenience. tion of the President's Office of Science and Technology and the Federal Council for Science and Technology, acting upon the recommendation of the Council's Committee on Scientific and Technical Information. The National Bureau of Standards has been assigned responsibility for administering the effort. The general object of the system is to coordinate and integrate existing activities in data evaluation and compilation into a systematic comprehensive program, supplementing and expanding technical coverage when necessary, establishing and maintaining standards for the output of the participating groups, and providing mechanisms for dissemination of the output as required.

The NSRDS is a decentralized operation of nationwide scope, with central coordination by the Bureau; it comprises a complex of data centers and other activities carried on in government agencies, academic institutions, and nongovernmental laboratories. The independent operational status of existing data projects is maintained and encouraged. Data centers that are components of NSRDS produce compilations of critically evaluated data, critical reviews of the state of quantitative knowledge in specialized areas, and computations of useful functions derived from standard reference data.

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# STANDARDS NEEDS OF A MAJOR NATIONAL AEC LABORATORY A. F. Rupp

At ORNL our need for standards is quite general. Being one of the largest nuclear research laboratories, our interest extends over the entire range of nuclear and radioactivity standards and includes, in addition, many other standards.

ORNL has a long history of not only making use of nuclear standards, but also of developing them. In the early days of operation of the Clinton Laboratories (which later became ORNL) there were very few generally accepted nuclear standards and knowledge of the characteristics of the many recently discovered radionuclides was very sketchy. Some of the earliest work was directed to the urgent problem of standards for radiation dose measurements in health physics work. Half-lives and radiation characteristics of many radioisotopes were measured and methods of measuring neutron fluxes for reactor control were among the first areas for concentration.

One of my own principal fields was radioisotope production and distribution in which the need for radioisotope assay standards was immediately apparent. For a number of years, ORNL served as an unofficial source of standards for the assay of most of the radioisotopes we distributed, even though at no time was an explicit or even implied certification furnished with radioisotope shipments. Intra-lab standards were developed to bring harmony to the comparison of results within the Laboratory and as absolute counting improved, and better means of measuring photon energies became available, new and better decay schemes were issued. Many other laboratories in the U. S., including the National Bureau of Standards, were doing similar work and samples were interchanged to get comparative measurements. The National Bureau of Standards issued its first calibrated standard, for other than radium, <sup>131</sup>I, in Active cooperation has been maintained between ORNL and 1948. NBS, in which we engage in cross-checks on round-robin samples and furnish ultra-pure radioisotopes for the preparation of

### ORNL-National Laboratory

samples and standards. Similar cooperative efforts between ORNL, the NBS, and many other government, university, and commercial laboratories has continued in all other phases of radiation and nuclear standards, including participation with the IAEA in establishing international standards.

Management, as a rule, has little direct contact with the technical aspects of the establishment and use of standards, but has come to regard standardization as an accomplished fact. For example, in reading a report giving the statistics on radiation exposure to workers in the various nuclear plants and laboratories around the world, no need is felt to have the figures checked and the results normalized. Radioactive materials being sold to other laboratories will be measured against the same standards in the purchaser's laboratory as those used at ORNL. Safety information on the control of reactors can be translated from one reactor installation to another with reasonable surety. These are the kinds of things that management is primarily concerned with - the smooth interaction of an establishment within itself and with other institutions throughout the world.

At ORNL we make great use of standards of all kinds and indeed, nuclear standards are only a part of the many standards in daily use - ranging from standard electric potentials to accurate weights for cable-testing. Among direct nuclear standards, those used for assay of radioisotopes and calibration of radiation detection instruments are probably the most important. We purchase standards from a number of organizations: NBS, IAEA, U.K. Radiochemical Centre, U.S. commercial laboratories (secondary standards checked against NBS, and used for assay calibration by many of our radioisotope customers). We have found agreement among all these various standards to be quite close ( $\sim 2\%$ , 1% from the mean), with the exception of occasional discrepancies we have noted in some commercially available standards.

Procedures for radioisotope analysis and assay have been collected in the ORNL Master Analytical Manual (TID-7015, 1957) which is constantly kept up-to-date by supplements.

#### Rupp

Standard materials, methods, and data are important in work with neutrons, x-ray and gamma radiations. We follow ASTM, USASI or IAEA recommendations whenever applicable, and many of our people work with the groups developing these standards.

Standards for fast neutron measurement in evaluation of damage to materials by fast neutrons are needed. The development of large isotopic power sources has brought about a need for accurate calorimetry standards, and reliable data on the relationship between the heat-power output of radioisotopes and the output as calculated from the decay scheme. The large isotopic power sources such as <sup>90</sup>Sr titanate generate heat by the internal absorption of beta radiation and some new information on the integrated beta spectrum has been noted in making comparisons between the radioisotope assay and the heat output. Quite possibly standard calibrated isotopic power sources may be developed eventually that would be quite useful if the calorimetric method is used for assay of kilocurie or megacurie quantities (and it is quite likely that the calorimetric method will be the only good method available).

Speaking of beta sources, there is need for developing calibrated, high-intensity, relatively long-lived NBS certified beta sources of 10 - 1000 r/hr. Probably  $^{90}$ Sr would be the principle radioisotope to be used for such sources, but  $^{147}$ Pm, and  $^{171}$ Tm would also be useful for lower energy standards.

Accurate compilations of nuclear data are essential for reference in a big nuclear research laboratory. Examples are the <u>Table of Isotopes</u> by the Berkeley group, the journal <u>Nuclear Data</u> now being put out at ORNL and the "Barn" book put out by Brookhaven. These essential data collections are "standards" of a sort and need only to be improved by faster means of keeping data current.

ORNL has a large project for evaluation of reactor materials, quality assurance, operational testing and engineered safeguards which utilizes many standards in addition to those commonly referred to as nuclear standards. These include the metals, both ferrous and non-ferrous, the primary chemical standards, ceramics, etc. Progress in nuclear science and technology depends on good standards. Those who request them and those who prepare them have a great responsibility. Preparation requires exacting work and a great deal of money. The only thing worse than no standard at all is a poor or unreliable standard.

# NUCLEAR STANDARDS AND THE AEC NEW BRUNSWICK LABORATORY C. J. Rodden

Of the many meanings of the word standard that one finds in the dictionary the one that in my opinion more clearly defines its use by the chemist is "something that is set up and established by authority as a rule for the measure of quantity, weight, extent, value or quality." Under this definition there are two possibilities that one should con-First is that we are going to have a material which sider. is well documented as to the value in question and the second is we have an accepted method of determining a certain constitutent. In the first category we have what is referred to as primary standard materials and also analyzed samples. In the second category we have standard methods of analysis such as one would find in the ASTM Methods for the Chemical Analysis of Metals, in the USA Standards Institute standards, or in other publications.

Let us now consider what is desirable in standards for chemical analysis.

It should be a metal, alloy, compound or ore which is homogeneous and is stable under all normal atmospheric conditions. Since we are dealing with materials which do not always follow this criterion, it is desirable that, if any change does occur--such as the absorption of water--the material can be brought to a certain composition by a standard heat treatment (the usual condition is to heat to constant weight at 105°C). In the case of the very reactive metals with which one deals in the nuclear energy field, one may be forced to use a chemical treatment, such as the pickling of uranium with nitric acid or the electropolishing of plutonium to remove an oxide coating. This is not ideal, but it may be the best that can be done. In certain other instances, materials of a hygroscopic or oxidizable nature are packaged into bottles in a dry box and sealed. Needless to say, this type of sample is not the most reliable. Human fallibility 227

must also be considered in the preparation of standards. There is always the possibility of mislabeling, of mixing samples or using unclean containers in the handling of the materials. All these must be guarded against. The amount of work that goes into the preparation of a reliable standard does not make this a very promising business venture.

In many respects the nuclear energy field is at a disadvantage since there are not enough nationally certified standards available. This has resulted in many companies setting up their own standards. This works very well in running a plant but may cause problems when inter-company or international transfers occur. It is highly desirable that certified standards acceptable to all parties concerned be available.

The New Brunswick Laboratory had its roots in the National Bureau of Standards and it is not surprising that from the first years of its operation standards have been considered at one time or another. We started to assist an embryonic industry. Volume was too small to interest the usual standard suppliers. These standards were broken into two types of materials. (1) Series of impurities contained in various matrices such as uranium oxide, beryllium oxide and thorium oxide for the use in spectrographic analysis of impurities in nuclear materials. (2) Another group was what would I presume would be called analyzed samples. One of these was  $U_3 O_8$  that had been analyzed under certain conditions by a considerable number of highly competent laboratories in the United States and which was distributed by the New Brunswick Laboratory for use in the analysis of uranium. I believe that in the analysis of any material it is much to be preferred to have a standard that is similiar in nature to what is being analyzed. In the determination of uranium I believe it is much better to standardize with uranium. In addition to these chemical standards certain analyzed samples have been distributed for use in radiochemical work. Most of these are used or have been used in the determination of ura-

#### Rodden

nium in ores and such materials. In addition to these there were samples of analyzed materials such as pitchblende and carnotite which were used for the determination of their uranium content and monazite for both thorium and uranium content.

Table I lists the chemical standard materials with which the New Brunswick Laboratory has been engaged. All uranium is of normal isotopic composition.

TABLE I

CHEMICAL COMPOUNDS

Samp	le No.	Purity	Analysis for
950a	NBS U308	99.94%	U
	U	99.97	U
17b	UF <sub>4</sub>	100.09	U, $U^{+4}$ , $U0_2$ , metallic
			impurities
18	UO3	98.67	U, H <sub>2</sub> 0 impurities
948	NBS Pu(S04)2.4H20		Isotopic composition
	Pu(S0 <sub>4</sub> ) <sub>2.4</sub> <sup>H</sup> 2 <sup>0</sup>		Pu

Early on in the uranium project U<sub>3</sub>0<sub>8</sub> was obtained and analyzed at the National Bureau of Standards and distributed by the Uranium Section. When we moved to New Brunswick this material was taken with us and distributed for quite a number of years from the New Brunswick Laboratory. New material was obtained when the first batch ran out. Several years ago this material was transferred to the National Bureau of Standards who now distribute the material under the No. 950a. It is the most widely used standard for the determination of uranium. Incidentally when the Mallinckrodt Chemical Co. at Weldon Springs went out of operation there were several hundred pounds of this material still in drums at that installation. I decided it would be better to save this material than to have it dumped into the regular scrap so this material is now at the New Brunswick Laboratory.

However, the fact that this  $U_30_8$  is only 99.94% pure does have some disadvantages;  $U_30_8$  is not easily obtained in stoichiometric concentrations. Quite a number of years ago

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we obtained at the New Brunswick Laboratory some dingot material from Mallinckrodt. This material is uranium that has never been remelted in graphite so the purity is considerably higher than the remelted material. For many years I have been a proponent of uranium metal as a standard for uranium rather than  $U_3 0_8$  because of this difficulty of the stoichiometry of U308. This dingot uranium metal which has been fairly widely distributed and analyzed throughout the world has a value of 99.97% for the uranium content. It is expected that before long this will be available for distribution. by the National Bureau of Standards. The UF, is a material which is used for assay purposes. It is still available from the New Brunswick Laboratory. The UO $_{3}$  (98.7%) is not a stable compound and it is only sold in sealed containers. As a primary standard it is not satisfactory, in fact it can only be used when initially opened since it will change in moisture content. This compound however has been fairly widely used as a base to make spectrographic working standards since it is quite readily ignited to  $U_2 O_8$ .

Two preparations of plutonium sulfate tetrahydrate have been made at the New Brunswick Laboratory. One of these was for isotopic composition the other was for use as a chemical standard, since the only other standard available for plutonium is one in which a weighed portion of metal has to be used in its entirety. These materials have been made for distribution by the National Bureau of Standards. In addition to this type of material other samples have been prepared at the New Brunswick Laboratory for use in various operations. In the early uranium rush in the United States a lot of people were interested in obtaining samples of ores of known uranium concentration to be used for chemical analysis. We obtained samples of carnotite, pitchblende, monazite sand and phosphate rock as is shown in Table II.

	ŗ	PABLE II	
	ANAL	YZED SAMPLES	
Sample No.	Material	U-08 Content	Th02 Content
1	Phosphate Rock	0.029%	-
3A	Pitchblende	4.29%	
4	Carnotite	0.18%	
5	Carnotite	0.11%	
6	Pitchblende Ore	e 53.5%	
7 A	Monazite Sand	0.4%	9.7%

These materials were analyzed by a group of laboratories around the United States and values were placed upon them. These are still distributed by the New Brunswick Laboratory. All of these materials are natural products with the exception of 3A pitchblende which was made by taking high grade pitchblende and grinding and mixing and blending it with dunite. This material is then analyzed before distribution.

While we were at the National Bureau of Standards requests were received for a series of graded samples containing uranium and thorium that were at equilibrium with their daughter products. In order to do this a sample of pitchblende was obtained, and analyzed for uranium and radium. It was shown to be at equilibrium. It was then ground and mixed with dunite which was radioactively inert. These materials, Table III, could then be used by people for calibrating counting equipment to be used in the field. A series was also made for thorium under the same method of manufacturing by diluting monazite with dunite. These have been used not only for counting purposes but have been used to a fairly large extent in chemical analysis. These samples were made however for use as counting standards and a fairly large sample was expected to be used. Of course occassionally somebody decides to use a ten milligram sample and in that case the values assigned to them may be considerably in error. In the past year or so due to the revised interest in uranium mining there has been an increase in the sales of the counting standards.

### TABLE III

COUNTING STANDARDS FOR COUNTING WORK

Sample No.	Material	<u>U</u> Content	Th Content
42-1 to 42-4	Pitchblende-dunite	4.0 to 0.5%	
73 to 77	Pitchblende-dunite	l.0 to 0.001%	1
79 to 84A	Monazite-dunite	0.04 to 0.00004%	1.00 to
			0.001%

From the very beginning of the uranium program back in the early forties the question of the impurities in nuclear materials was of the greatest importance. Since it was found out early that most of the impurities could be analyzed by spectrographic means series of graded samples containing a considerable number of elements was prepared. The three which were prepared and which are still available are uranium oxide, beryllium oxide and thorium oxide. These contain a varying amount of impurities which can be determined by spectrographic analysis. These graded series enable one to prepare curves for spectrographic analysis. They have been widely distributed throughout the world and are still distributed on a fairly regular basis. These materials, Table IV, were made synthetically.

# TABLE IV SAMPLES FOR SPECTROGRAPHIC ANALYSIS

Sample No.	Material					
66-1 to 7	Th02	Graded	series	for	22	elements
72-1 to 5	BeO	"	11	"	17	**
96-1 to 6	Be0	**	11	11	21	11
95 <b>-</b> 1 to 7	U <sub>3</sub> 08	**	TT	11	22	**

In addition to the spectrographic samples, we have distributed, at one time or another, metallic samples of uranium, thorium, and beryllium. The uranium and the thorium were generally in the form of machined chips while the beryllium metal was in the form of powder.

At the present time the beryllium is the only material that is available since the uranium has been depleted and we have not renewed this up to the present. We do have dingots

#### Rodden

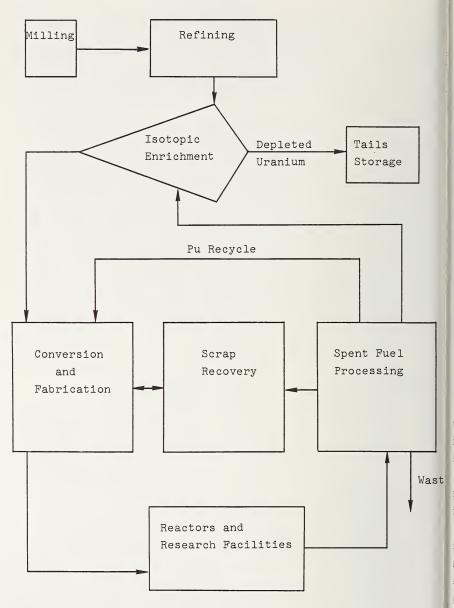
which can be turned down however for this purpose. The thorium metal chips were not very successful. The material oxidized quite rapidly and it was not satisfactory for use as an analyzed sample. These metal samples were blended in a mill and then analyzed to see if noticeable segregation was present in the material.

There is another service that the New Brunswick Laboratory performs but whether it comes under the heading of standards is a little uncertain. It is known as the General Analytical Evaluation Program. Figure I shows diagrammatrically the uranium-plutonium cycle. I believe it is selfexplanatory. The General Analytical Evaluation Programs are concerned with material at various stages of the cycle. The New Brunswick Laboratory distributes batch production and analyzed or synthetic materials on a regular basis to a number of cooperating laboratories who determine various elements or impurities as is indicated in Table V. Results are accumulated and statistically analyzed at certain periods.

# TABLE V

GAE	PROGRAMS
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Material	Analysis
U Concentrates	U <sub>3</sub> 0 <sub>8</sub>
UO <sub>3</sub>	U, Fe, Ni, Cr
UF <sub>4</sub>	U, U <sup>+4</sup> , Fe, Ni, Cr
U	Density, N, C, H, Fe, Si,
	Ni, Cr, Mn, B
UF <sub>6</sub>	U, U-235, impurities
Enriched U Scrap	U, U-235
Dissolver Solution	
Uranyl Nitrate Enriched	U, U-235, impurities
Plutonium Nitrate Solution	Pu, Pu-239, Pu-241
Plutonium-Uranium	Pu
Molybdenum Solutions	
Pu02-U02	Pu, U



# FIGURE I. URANIUM PLUTONIUM FUEL CYCLE

#### Rodden

It will be noted that certain portions of the entire cycle have been considered. In certain operations solutions are the material analyzed such as plutonium nitrate solution from the spent fuel process or dissolver solutions from enriched uranium cold scrap. In the scrap recovery program many types of solutions are obtained and synthetic solutions of a considerable number of these fuel materials have been distributed such as U-Al, U-Zr, U-SS, U-Mo, U-Mo-Zr, U-Ni and U-Be or oxides of the elements. In conjunction with this same type of work several of the manufacturing companies have asked us to make synthetic solutions for them that they can use in their analytical control work. So in a sense you can call these custom analyzed materials or what you wish. We anticipate that with the increase in the safeguards program that this type of work will increase over a large portion of the entire uranium-plutonium processing cycle. It may be of interest that just as I was writing the last sentence a request came in from one of the fuel manufacturing companies asking whether we knew where they could get an aluminumuranium standard for use in their plant. Since we did not know where such was available they proposed that we would make such a standard with them supplying the material. This is just an indication as to where we feel that the standards of this type are going. Little has been said about the spent fuel processing and in fact little has been done on actually checking of this type of material.

We are hoping that within a few months work will start at the New Brunswick Laboratory on the construction of a facility to handle irradiated dissolver solutions.

There is one other subject that I would like to touch on briefly, which I mentioned at the start when I talked about standards. It is the question of standard methods of analysis. If we refer to the uranium-plutonium fuel cycle you will see that we should have standard methods of analysis for many types of material in case of disagreement. It is preferable if such methods would come out under the aegis of

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some United States standard group. However, at the present time the New Brunswick Laboratory has the job of revising the "Selected Measurement Methods for Plutonium and Uranium in Nuclear Fuel Cycle". This revision, which will follow the ASTM format, is definitely a change over from the old one and is going to be tied to a very large extent to the cycle of the fuel element as it goes through the uranium and plutonium reactor cycle. This work is being done in cooperation with many of the laboratories in the United States who have had a long experience in the analysis of this type of material. It is hoped also that in the not too distant future that these methods of analysis can be taken by one of standards group and made official US methods.

# THE NEED FOR STANDARDS IN ENVIRONMENTAL ANALYSIS John H. Harley

The radiochemical analysis of environmental samples covers a broad spectrum of sample types and an even larger number of radionuclides. This is true whether the analyses are conducted for surveillance or for research and whether the radionuclides involved are from weapons test fallout, from industrial releases, or are naturally occurring.

As you are all aware, very few samples can be counted on an absolute basis, therefore it is necessary to have instrument standards for calibration. Bill Marlow has shown some tables indicating the nuclides that are presently required as standards for the Health and Safety Laboratory and I am sure that other groups such as the National Center for Radiological Health would have very comparable lists. These standards should normally be known to a few percent, even though the required precision or accuracy of the analyses may not be that high for surveillance. Many of the research or development studies require better data for direct scientific purposes.

While the need for extreme accuracy in the standards is not high there is a need for reliability. There are very few fields of analytical chemistry where individual results may be subject to such public scrutiny, including even congressional investigation. It would be desirable to have an objective government body stand behind these standards. The few nuclides now available from the Bureau of Standards are satisfactory but then we have also had good results from the International Atomic Energy Agency and the Radiochemical Center at Amersham. With these latter two groups we do checks on their accuracy and with few exceptions they have met specifications. There are a number of nuclides that are not available as standards from any of these groups and it is therefore necessary for us to maintain our own capability for standardization.

### Standards-Environmental Analysis

Up until now I have been speaking specifically of relatively high activity standards needed for instrument calibration. There is another type of standard material which is also needed. Our laboratory attempts to maintain a quality control program in which about 15% of the analyses are split samples, standards or blanks. Thus we need bulk samples containing the nuclides in question in a matrix which is directly comparable to the samples being analyzed. In our case the samples are analyzed blind and therefore the quality control samples must resemble the working samples very closely. This is only possible by having the chemist receive ashed material rather than the original samples.

This type of standard must be prepared in our laboratory. The starting materials may be several bags of grain, several hundred pounds of dried milk, or hundreds of pounds of bone. In all cases these must be ashed and blended to prepare homogeneous materials which can be used for quality control over a period of time. For standardization we use the technique of the National Bureau of Standards, that is asking competent laboratories in the field to analyze the material and then setting up a standard value from the data received. This operation is probably not a fruitful one for a central standards organization since the need is limited to relatively few laboratories.

In conclusion I would hope that we will have available reliable standards for at least our instrument calibration. It seems to me that preparing these standards is a difficult way to make money and that it would have to be subsidized by the government.

### PANEL DISCUSSIONS

### J. P. Cali and H. F. Beeghly

Four distinct and separate panel discussions were held during the course of the Symposium. They were: 1) ACS Representations on USASI "N" Committees; 2) NBS Standard Reference Materials for the Nuclear Field; 3) Present and Future Needs of the AEC for Standards for Nuclear Chemistry and Technology; 4) Unfulfilled Present Needs and Future Demands for Standards for Nuclear Chemistry and Technology. Unfortunately, because the decision to publish the proceedings had not been made prior to the Symposium taped remarks were not made of these discussions and only notes were available from which this section is compiled.

### FIRST PANEL DISCUSSION

The first panel on ACS Representatives on USASI "N" Committees heard various members of eight USASI "N" Committees describe briefly the role of each committee in the nuclear field. Representative of the presentations made are two following short summaries prepared by B. M. Robinson and A. Glassner.

# SUMMARY OF USASI N-14 COMMITTEE ACTIVITIES B. M. Robinson

N-14 "Packaging & Transportation of Fissile & Radioactive Materials" evolved from the former ASA N 5.5 committee. The new group held an organization meeting in December, 1957 followed by sessions in March and July.

The scope of the N-14 committee may be summarized as: "Standards" for the packaging and transportation of fissile and radioactive materials but not including movement or handling during processing and manufacturing operations.

At the recent San Francisco meeting in July, Committee Chairman, Roger Waite of the American Insurance Association, cited the following objectives for the Committee:

- (1) Comment on DOT Regulations.
- (2) Develop Guides to the Regulations.

- (3) Explain the Regulations.
- (4) Develop requirements and recommendation going beyond the Regulations that will be of help to industry.

The Committee includes personnel from the nuclear industry, both government and private, the regulatory agencies, the insurance field, and the carriers. It is a blue ribbon group since the people involved are experts in the field. The N-14 Committee furnishes representatives for the International Standards Committees.

The N-14 Committee has been divided into the following sub-committees:

- N14.1 Fissile Materials (small sources) not calling for special shielding, and "type B" packages for plutonium. Chairman: W. A. Smith, Jr., National Lead Co.
- N14.2 "Type B" packages (moderate to large sources non-fissile isotopes and irradiated fuel (divided into five additional sub-committees as listed below). Chairman: John W. Langhaar, duPont Co.
- N14.2.1- Develop a guide to help explain and clarify existing regulations. Chairman: C. W. Smith, General Electric Co.
- N14.2.2- Develop a guide to design features and fabrication methods which are suggested to comply with the regulations and methods of demonstrating compliance with the regulations. Chairman: R. W. Peterson, National Lead Co.
- N14.2.3- Develop standards for design and performance features of containers that are in addition to those required by regulations, either for safety or for uniformity of handling and operation. Chairman: E. C. Lusk, Battelle Memorial Institute.

### Cali, Beeghly

- N14.2.4- Cask tiedown committee. Chairman: J. W. Langharr, duPont Co.
- N14.2.5- Guide for administration and handling including special permit procedures, contamination control, insurance, placarding, etc. Chairman: W. R. Romine, Dow Chemical Co., Rocky Flats.
- N14.3 Low specific activity non-fissile materials, including bulk materials. Chairman: Alexander Aikens, Jr., Capintec, Inc.
- N14.4 Small sources of non-fissile materials, including "Type A" packages. Chairman: Leonard Horn, Underwriters' Laboratories.
- N14.5 Transport through tunnels, bridges, and toll roads. Chairman: Frank Sweeney, Associated Transport, Inc.
- Ad hoc Subcommittee Compendium of container tests. Chairman: E. C. Lusk, Battelle Memorial Institute.

Even though only recently created, the N-14 Committee promises to fill a very important niche in the nuclear industry which in a relatively few years will be one of the largest in the world. The amounts of material to be moved will be steadily increasing and many shipments are involved.

We are in a transitory stage with respect to both domestic and international shipping regulations. In the U. S., the Department of Transportation is now the centralized authority on shipments with existing permits expiring and DOT taking over the responsibilities previously centered in other agencies.

Accordingly, N-14 can serve as a very effective communication medium since it brings together individuals from all facets of the nuclear industry involved with transportation. In addition, it will perform a task for both industry and the government in augmenting mechanics which otherwise probably would be decentralized and now can be directed to common goals. I am sure the N-14 committee was an important factor in the notice filed in the Federal Register three weeks ago by the DOT which expresses a difference in philosophy from previous concepts on regulations.

The Hazardous Materials Regulations Board (under DOT) plans to revise the regulations governing transportation casting them in general terms and eliminating much of the detail.

Performance standards are emphasized in contrast to manufacturing specifications.

Many of the N-14 committee members will be appearing on the program at the 2nd International Symposium on Packaging and Transportation of Radioactive Materials being held in Gatlinburg October 14-18, 1968.

# SUMMARY OF USASI N-12 COMMITTEE ACTIVITIES A. Glassner

In 1966, the USA Standards Institute (USASI) became the successor to the American Standards Association (ASA). In the reorganization that ensued, the ASA Sectional Committee was replaced by the N12 Committee, entitled "Terminology, Units, Symbols, Identification and Warning." The scope of the committee's work includes: (i) standards for nomenclature, definitions, and units; (ii) identification means such as symbols, signs, labels, or color codes; and (iii) warning means or devices, all involving nuclear and radiation activities. Membership of the committee consists of representatives appointed by many of the professional societies and a few industrial participants.

The principal work carried on by the new American Chemical Society representative, who has replaced Dr. W. Wayne Meinke on N12, has been with the subcommittee charged with revision of the ASA N1.1 <u>Glossary of Terms in Nuclear Science</u> <u>and Technology</u>, published in 1957. This has now been replaced by a new edition: USASI N1.1-1967, with the same title.

Terms in the new edition that were assigned to the American Chemical Society for proposed definitions were prepared with the help of a number of chemists throughout the country who are expert in the relevant fields. Inasmuch as the subcommittee has worked very closely with the corresponding group in the International Standards Organization (in fact, the USA delegation serves as the Secretariat of that group), some minor modifications of the proposed definitions occurred, principally of a semantic nature, in order that international agreement on definitions could be obtained. This work is still being carried on, and it is hoped that a much larger, internationally accepted glossary can be issued.

At this time it appears that new work of the N12 Committee will emphasize the preparation of standards for units and symbols.

#### SECOND PANEL DISCUSSION

The second NBS panel discussed various Standard Reference Materials now in process which will further standards work in nuclear technology. Much of the discussion centered around radioactivity standards issued both by NBS and industry.

The NBS panel discussed various Standard Reference Materials now in process which will further standards work in nuclear technology. Much of the discussion centered around radioactivity standards issued both by NBS and industry. There was considerable comment by the audience that a fair percentage of commercially available radioactivity standards were not sufficiently accurate for many applications. NBS was urged to expand its inventory to include many more SRM's than are now presently available, but it was pointed out by the NBS representatives that resources were exceedingly tight, and that without additional support such a course was presently unlikely.

#### THIRD & FOURTH PANEL DISCUSSIONS

Many of the questions raised during the third panel discussion were reiterated and further amplified in the fourth and final panel which was by far the most comprehensive and in essence summarized the entire symposium. For this reason, we will use the remaining space to discuss the final session in more detail.

In starting the last panel discussion, Dr. Meinke placed before the audience the following problems which were then discussed serially by the panel.

- The need for standards; their relative importance; the quantity -- are there enough? These questions were disuessed from four aspects: legal, safety, technical, and administrative.
- (2) The speed (or lack thereof) with which standards needs are identified and then produced; time schedules; timeliness.
- (3) The problem of proliferation of standards and standards committees and organizations; overlapping.
- (4) Liaison with international standards organizations.
- (5) Realistic criteria; responsible standards.
- (6) Private sector-government interactions.

Dr. Meinke in his opening remarks also pointed out that the panel members had been chosen so that representatives of the following interested groups would be present: standards organizations (USASI, ASTM); government (AEC, NBS); associations (AIF, ANIM); industry (Westinghouse, Nuclear Chicago, etc.); and, the laboratories (ORNL, Battelle).

In the discussion of item 1, above, the point was strongly made that safety and technical considerations very often cannot be separated or discussed in the context of one or the other alone. In some cases in the past it was felt that safety may have been overemphasized to the extent that technical goals were not realized. A further point made was that reliability and safety are not usually synonomous and should not be confused.

An NBS representative discussing the question of the number of standards pointed out that the availability of a

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wide range of SRM's could be met only when there was widespread demand and support(including financial) from outside groups. Very often in the past this has not been the case.

During the sessions there was general agreement that the total number of standards of all kinds, SRM's, standard methods, standard procedures, etc. were too few. Only in the area where legal requirements are set forth were there nearly adequate standards. It is obvious that a legal requirement almost automatically sets mechanisms in motion that produce standards.

In the field of safety standards, the situation is often very confused. There are at present several designs for pressure containment vessels and 70-80 vessels under construction. While there are no legal requirements for standards, from the stand point of safety these are required, but in fact do not now presently exist.

In the field of safety standards, the situation is often very confused. There are at present several designs for pressure containment vessels and 70-80 under construction. While there are no legal requirements for standards, from the stand point of safety these are required, but in fact do not now presently exist.

The question was raised as to the extent government should be in the standards business. Two points were in general agreed on: where legal requirements exist, then the regulatory bodies (AEC, e.g.) have no choice in the matter and standards become an important aspect of their activities. The AEC representative did emphasize, however, that the AEC is very happy when standards are developed voluntarily by industry, or standards bodies, but he also said that there always comes that point in time when it is essential that a standard be on the books, and if industry or a standards group has not met a schedule, then the government must step in by default. The second point was that for the U. S. the present system of largely voluntary standardization was much to be preferred and,

#### Panel Discussions

indeed, encouraged as opposed to legally imposed and nonvoluntary standards.

There was a consensus that both the timeliness and speed with which standards were produced left a great deal to be desired. Each group responded affirmatively to the query put by the moderator on the question of the desirability of speeding up the standards process. On the point of whether the industrial and government groups would be willing to help finance steps which would speed up the process, there was some feeling that additional dollars might be forthcoming, but, of course, no definite commitments were asked for, only more or less personal expressions of possibility and feasibility.

Mr. Caum, ASTM, stated that because of the large paperwork load placed on the ASTM Committees' secretaries, that the ASTM was starting to employ full-time personnel for these tasks. With the reorganization of ASA into USASI, Mr. Chalker pointed out that the base of support had been broadened to include government agencies and other groups so that some of the more time-consuming procedures hopefully could be shortened.

J. W. Crawford of the Reactor Technology Division, AEC, said that his Division was highly involved in support of standards work. He felt he had to be quite careful in choosing his words as to the additional support his Division could make, but said that if there were additional standards problems in areas of pertinence, he would be interested in hearing about them.

A. R. Van Dyken from the Research Division (AEC) next pointed out that work has been sponsored in standardization of very low levels of radioactivity in contamination of materials as well as in certain other areas. However, standardization becomes most pertinent and important once problems have progressed from the research stage to the development or production stage. Much of the standardization of pertinence to the AEC is done in some of the other Divisions such as Safeguards, Isotopes Development, Biology and Medicine.

Leroy Nauderhaug, speaking for Safeguards, (AEC) reviewed work going on in standardization in a number of areas and remarked that if there were additional problems in these or related areas that his Division would be interested in discussions regarding them. Bill Marlow from Biology and Medicine (AEC) also concurred and said his Division would be amenable to discussions for support for standardization work. During the two days of sessions it had become apparent that one of the major problems was the production of standardized radioisotopes for health and safety types of measurements. In a number of cases there was overlapping work being done in different AEC Laboratories, because there was no centralized place where standards could be obtained. Finally, Warren Eister from Isotopes Development (AEC) discussed the support they were already giving to standardization and expressed interest in continuing support in other problem areas.

From the National Bureau of Standards point of view W.W. Meinke said that at the present time, while the Bureau did have the personnel, equipment, and facilities to broaden their program in radioactive materials, that because the program was not at present on a self-sustaining basis, any expansion would have to be covered by outside support. It was pointed out that during last fiscal year the Bureau invested twice as much support in the radioisotope standard materials program as was returned through sales. On the other hand, should outside support be forthcoming then NBS is presently in a position to expand considerably its radioisotope standard materials program. Wilfred Mann and Sam Garfinkel (NBS) agreed with this presentation and analysis.

Finally, as a spokesman from the national laboratories, Art Rupp agreed that if no one else did the standardization work and a standard became essential for a particular laboratory program, that individual laboratories had to put forth the effort. He did point out, however, that the laboratories have funding problems also and must gain support for such efforts. After this long discussion of further and/or additional financial support, the remaining time was spent in a discussion of the remaining items on the agenda.

On the question of the speed of production of standards, D. Sunderman stressed the point that in standards work very often a certain induction period is required and therefore in some cases it will not be possible to reduce the time to a few months. In contrast to this view, several others gave examples, where, when the lack of a standard was seriously holding up progress, a group working very intensively did in fact produce a standard ready for the consensus' opinion in a few months. There was a general feeling that the consensus operation was the time lengthening factor in many cases, but most agreed that the consensus principle was too important to tinker with seriously.

On the question of the proliferation and overlapping of standards, J. Caum, ASTM, reported that the present horizontal structure of the ASTM is a recognized weakness leading to the duplication and overlapping of standards. Reorganization is now underway and will be along the lines of products/industry groupings. R. Chalker, ANS, believes that the close involvement of his society with USASI is the best means of ensuring good, pertinent standards, and the best means of avoiding duplication. Mr. Chalker recommended that compilations, similar to the ORNL publication on nuclear standards groups, be made to show which group, committee, or organization is working on a particular standard or group of standards. He also stressed that a small concentrated group is best for actually writing the standard, then a large group is brought into the picture for the consensus process.

It was apparent from the lack of discussion that liaison with international organizations is not strong, although certainly not completely absent. W. Mann, NBS, stressed the importance in his field of supporting and continuing the work of international comparisons. This work is certainly in the

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strong interests of the United States. Also from NBS, Dr. Meinke told the symposium that his organization is to become more heavily involved in the international aspects of standard reference materials. This objective has been laid down by the Director, but plans for implementing this goal are not yet firm.

J. Kelly, Westinghouse Electric, was of the opinion that the most realistic criterion to use as a basis for standards was economic in nature, that standards so based were often the optimum standards. J. Crawford, AEC, said that many of the most realistic standards of recent years were those which relied heavily on past history and practice.

Finally, the discussion ended with a few brief remarks on the private sector-government interaction. The AEC, reported J. Crawford, cooperates with industry primarily through its membership on the Nuclear Standards Board of USASI. One AEC representative is always on the executive committee. In addition, the AEC is well represented on ANS, AIME, and ASTM standards committees. Mr. Chalker, ANS, asked that standards be formulated by workers and users in the field. These need to be more pragmatically oriented, less analytical, and only after the standards are well-established and agreed on that the legal regulations be established.

The discussion ended on this remark by E. Wiggin of the Atomic Industrial Forum, that there has been more government regulation recently, because, in his opinion, industry has neglected its role of providing standards.



#### APPENDIX

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